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Test Program to Demonstrate the Stability of Hydrazine in Propellant Tanks

Final Report

Clifford M. Moran Roy A. Bjorklund



April 1983

Prepared for

United Kingdom Treasury and Supply Delegation Washington, D.C.

through an agreement with

National Aeronautics and Space Administration

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Jet Propulsion Laboratory
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ABSTRACT

This document reports the analyses and results of a 24-month coupon test program to evaluate the decomposition of hydrazine by metallic components of propellant tanks. The propellant fuel evaluated was monopropellant-grade hydrazine (N_2H_4), which is normally a colorless, fuming, corrosive, strongly reducing liquid. The degree of hydrazine decomposition was determined by means of chemical analyses of the liquid and evolved gases at the end of the test program. The experimental rates of hydrazine decomposition were determined to be within acceptable limits.

The propellant tank materials and material combinations were not degraded by a 2-year exposure to hydrazine propellant. This was verified using change-of-weight determinations and microscopic examination of the specimen surfaces before and after exposure, and by posttest chemical analyses of hydrazine liquid for residual metal content.

ACRONYMS

ACS attitude control system ARDE ARDE, Inc., Mahawah, NJ

BAT Bell Aerospace Division of Textron, Inc., Buffalo, NY

BE binding energy

Caltech California Institute of Technology

CRES corrosion-resistant steel
CPR coupon preparation requirement

EB electron beam

EPR ethylene propylene rubber ETS Edwards Test Station, JPL

FEP fluorinated ethylene propylene (Teflon)

HAS hydrazine actuation system

HAZ heat-affected zone

JPL Jet Propulsion Laboratory

NASA National Aeronautics and Space Administration
OAST NASA Office of Aeronautics and Space Technology

OSS NASA Office of Space Sciences

PES photoelectron spectra

SEM scanning electron microscope
STP standard temperature and pressure
to be defined/determined/done

TIG tungsten inert gas

UKTSD United Kingdom Treasury and Supply Delegation, Washington, D.C.

XPS X-ray photoelectron spectroscopy

SECTION I

INTRODUCTION

A. COUPON TEST PROGRAM

The Coupon Test Program has been an investigation of the reactive compatibility of hydrazine with various metallic components of a propellant storage tank. The hydrazine/material compatibility research reported here was performed by the Jet Propulsion Laboratory (JPL), California Institute of Technology (Caltech), under Contract NAS7-198 with the National Aeronautics and Space Administration (NASA) for the United Kingdom Treasury and Supply Delegation (UKTSD) in accordance with the UKTSD Letter Agreement F-2479, dated July 5, 1979.

This coupon test program is an extension of the ongoing JPL/NASA long-term propellant/material compatibility program. The same procedures, test methods, and test facilities developed under the JPL/NASA program have been applied to this program.

This document is the final report for the Coupon Test Program. An interim report from Program A was prepared in October 1981 (Reference 1).

B. BACKGROUND-RELATED TECHNICAL WORK

JPL has collaborated with other agencies on a variety of research, development, test, and evaluation projects. The laboratory, with its Pasadena facility and Edwards Test Station (ETS) at Edwards Air Force Base, California, maintains an institutional capability and technical expertise in evaluating and testing Earth— and space—storable liquid propellants and materials for space—craft propulsion system applications. Specifically, JPL has been investigating material compatibility involving Earth—storable propellants, including hydra—zine, since 1962 under sponsorship of the NASA Offices of Aeronautics and Space Technology (OAST) and of Space Sciences (OSS). The details of the JPL material compatibility program and interim experimental results of the long-term storage testing are reported in References 2 and 3. The long-term exposure testing continues, and the accumulated time for some test specimens exceeds 12 years.

The results obtained have provided reliable data for designing and qualifying chemical propulsion systems and components for long-life spacecraft. The work performed has directly supported the early JPL planetary flight projects such as Ranger, Surveyor, and Mariner, and the Viking 1975 and Voyager 1977 (Jupiter-Saturn-Uranus).

The general technology areas involved are propellant chemistry, metal-lurgy, long-term (10-year) propellant/material compatibility, metal fracture/ toughness characteristics, and fracture mechanics design of pressurized systems. Typical Earth-storable propellants are hydrazine, refined-grade hydrazine (monopropellant grade), hydrazine-hydrazine nitrate, monomethylhydrazine, and nitrogen tetroxide. Spacecraft propulsion system materials include aluminum alloys, corrosion-resistant steels (CRES), titanium alloys, and elastometric materials. for example, AF-E-332.

C. OBJECTIVES OF THE COUPON TEST PROGRAM

The overall objective of the coupon test program was to verify the long-term compatibility of hydrazine actuation system (HAS) propellant tank materials and other material combinations with monopropellant-grade hydrazine. To accomplish this overall objective, the program was divided into two parts.

Program A was intended to evaluate short-term compatibility of the secondary propellant containment system shown in Figure 1-1. It should be noted that the secondary containment system will be exposed to hydrazine only if there is leakage from the primary containment system. The program objectives were:

- (1) Determine rates of hydrazine decomposition at 43°C by means of pressure rise monitoring throughout the term of the test program.
- (2) Verify that pressure containment materials and material combinations are not degraded by 6-month exposure to hydrazine propellant, using weight determinations and microscopic examination of specimen surfaces, after exposure.

Program B was intended to evaluate long-term compatibility of the primary propellant containment system shown in Figure 1-1. The program objectives were:

- (1) Determine rates of hydrazine decomposition at 43°C and 60°C by monitoring pressure rise throughout the term of the test program.
- (2) Determine degree of hydrazine decomposition by means of chemical analysis of liquid and evolved gases at the end of the test program.
- (3) Verify that primary containment materials and material combinations were not degraded by 2-year exposure to hydrazine propellant, using weight determinations and microscopic examination of specimen surfaces, after exposure, and also by posttest chemical analysis of hydrazine liquid for metal content.

D. MATERIAL COUPON SOURCE

The material coupons used in this program were provided by the UKTSD. They were obtained from sections cut out of a HAS tank fabricated by Bell Aerospace Division of Textron (BAT). A total of 82 coupons from 26 different locations are listed in Table 1. The locations on the tank from which the coupons were cut are shown in Figure 1-2. Each coupon was processed, weighed, cleaned, and individually sealed in a plastic bag by BAT before delivery to JPL. The coupons remained sealed until they were removed and placed immediately into glass capsule test units prepared at JPL.

¹All tables are contained in Section V.

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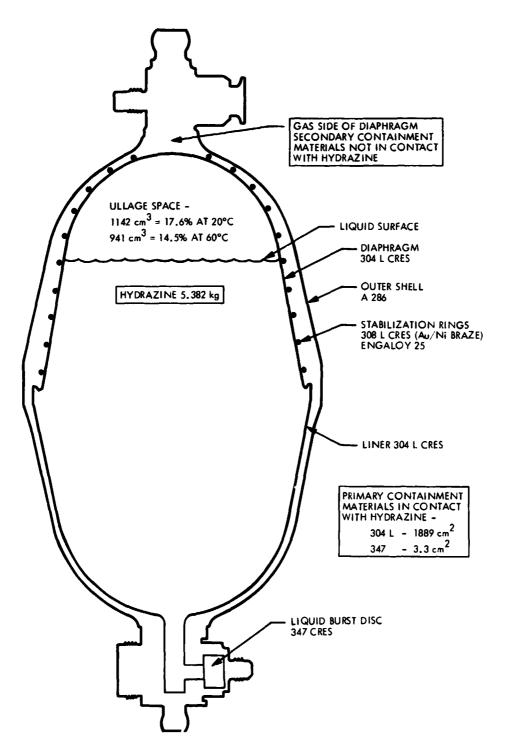


Figure 1-1. Hydrazine Actuation System (HAS) Propellant Tank Configuration

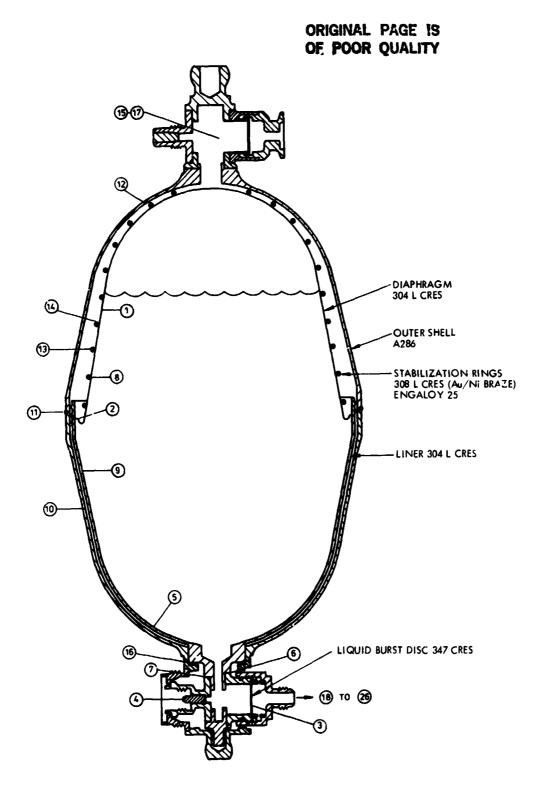


Figure -2. Coupon Location on HAS Tank

SECTION II

TEST PROCEDURES

A. TEST UNIT PREPARATION

Eighty-two hermetically sealed, glass-encapsulated test units were prepared by JPL with the materials specified and in accordance with the priorities established by the sponsor. All units were prepared in accordance with the procedures shown in Figure 2-1.

The test requirements for this program were specified in JPL Proposal 90-965, Revision 2, October 11, 1978 and are summarized below.

- (1) Purified hydrazine (VL-75 grade) was supplied by JPL. Pretest analysis of this propellant s shown in Appendix A. This propellant met the BAT material specification (Reference 4).
- (2) All test coupons were supplied by UKTSD, together with appropriate documentation prepared by BAT. For Program A, 38 coupons were designated; for Program B, 44 coupons were designated. The distribution of coupons by test storage temperatures was also designated.
- (3) The test containers were Pyrex capsules, as described in Reference 2, and designed to have an internal volume, when sealed, of about 80 cm³. Figure 2-2 is a photograph of a typical test unit.

Test equipment, instrumentation, and techniques duplicated those employed in the JPL 10-year test program and reported in Reference 2. The pretest procedures are summarized below.

- (1) Strain gauges used to measure internal pressure buildup were mounted on the open capsules. A preliminary pressure calibration was used to check the sensitivities of the strain gauges.
- (2) Test specimens were installed into the clean capsules in "as received" condition from BAT except for the EPR-515 O-rings which were coated with Krytox lubricant by JPL.
- (3) Funnel necks were fused onto the capsules, with care taken not to overheat the strain gauges. Final pressure calibration of the gauges was then made.
- (4) Internal volumes of the test capsules were measured by the expanding volume technique using high-purity gaseous nitrogen at ambient temperature.
- (5) Capsules were then loaded with enough hydrazine so that the combined volume of propellant and specimen was 40 ±0.5 cm³, and the specimen was fully immersed. Three of the specimens were found to be oversized and additional quantities of hydrazine were added.

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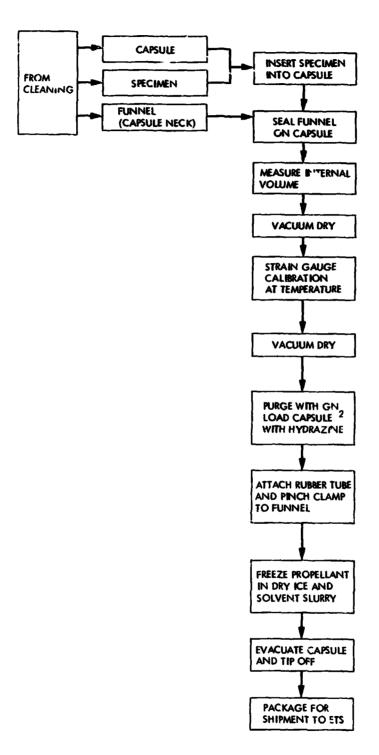


Figure 2-1. Procedures for Capsule Filling



Figure 2-2. Typical Glass Capsule Test Unit

- (5) The hydrazine was frozen in a dry-ice/alcohol slurry and the capsules were pumped down to a pressure of less than 1 mm Hg.
- (7) The capsule necks were sealed off by fusing the glass tube, and the completed test units were made ready for shipment to Edwards Test Station (ETS).

The hydrazine was pretest-analyzed for purity in accordance with the JPL standard methods (Reference 2). A special CO₂ analysis of hydrazine in one test unit, representative of a typical encapsulation, was performed to verify the exclusion of air during filling. (See Appendix A). No examination or analyses were performed on the coupons.

B. STORAGE TESTING

The experimental storage phase was conducted at ETS to determine the compatibility of the hydrazine propellant with the materials. A photograph of the "Lazy-Susan" type of storage facility is shown in Figure 2-3. The exposure tests on the 82 units were conducted for a period of up to 24 months (730 days) at temperatures of either 43 \pm 3°C (110 \pm 5°F) or 60 \pm 1°C (140 \pm 2°F). The temperature of 43°C was used in the JFL long-term program (Reference 2) as the "normal" temperature of a noncryogenic propellant in space. The higher temperature was chosen as being the highest temperature likely to be experienced by the propellant in service. During the 24-month exposure term, the following was accomplished:

- (1) Pressure readings were taken once per week for the first month.
- (2) Pressure readings were taken once per month for the next five months.
- (3) Pressure readings were taken bimonthly for the remaining eighteen months.
- (4) Test units were visually inspected after taking the pressure readings.
- (5) All results were recorded for the above observations.

Details of all test units are presented in specimen logs in Appendix B.

C. POSTTEST ANALYSIS

1. Discussion

At the completion of the storage tests, all test units were analyzed in accordance with the JPL standard methods (from Reference 2). The capsules were opened and the decomposition gases, hydrazine, and coupons removed. The test coupons were weighed and their surfaces were visually examined at 50x magnification. The decomposition gases and hydrazine were analysed using posttest procedures developed and used in JPL's original program. The procedure

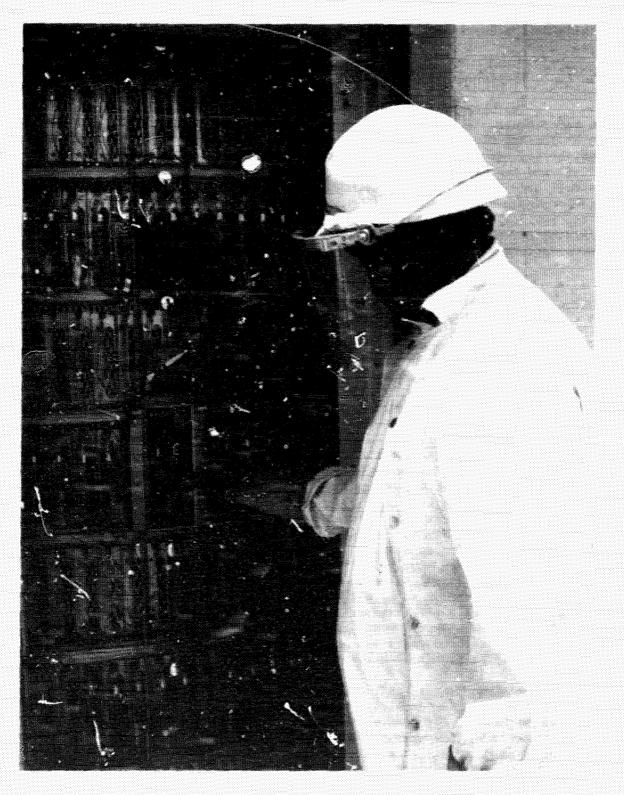


Figure 2-3. "Lazy-Susan"-Type Storage Facility

(Figure 2-4) provides for an analysis of all components. The choice of the posttest analyses to be applied is dependent on the test-unit history and the level of information required to permit satisfactory assessment of results. Details of the posttest analysis procedure are discussed in the following subsections.

2. Procedure - Complete Analysis

The hydrazine was frozen by immersion in liquid nitrogen (LN₂), and the capsule placed in the opening fixture (Figure 2-5). The capsule tip was broken and the volume of noncondensable gases (N₂ and H₂) measured in a calibrated vacuum system. The hydrazine was thawed and refrozen at -30° C (-22° F) and the gas at that temperature, mainly NH₃, measured. The residual hydrazine was removed using a syringe and the purity determined by a gas-chromatographic technique which measures NH₃ and H₂O. Metal content in the residual hydrazine was analyzed by atomic absorption techniques. A turbidimetric method was used for low concentrations of chloride; higher concentrations were titrated. Fluoride was determined colorimetrically.

3. Procedure - Limited Analysis

This procedure measures only the noncondensable gases, hydrogen and nitrogen. After thawing, the residual hydrazine was removed from the capsule using a syringe and was analyzed by gas chromatography for NH₃ and H₂O. If the NH₃ content is low, there may be an error due to NH₃ evolution before analysis.

4. Gases of Decomposition

a. Composition. The contents of the posttest capsule were frozen in liquid nitrogen and then prepared for sampling as follows: The strain gauge was very carefully scraped off with a sharp razor blade, a small scratch was made on the neck of the capsule, and the capsule was then enclosed in the opening fixture (Figure 2-5). The system was pumped down for several hours until moisture was removed from the outside of the capsule. The fixture was then filled with dry helium to 0.5 atmosphere to aid in heat transfer, and then immersed into liquid nitrogen to a depth equal to one-half the length of the capsule. After an hour, the helium was pumped out. When a satisfactory vacuum had been attained (1.3 x 10^{-2} N/m²), the gas sampling system was isolated from the vacuum pump, and the neck of the capsule was broken by turning the handle on the fixture. By means of a Toepler pump, the released noncondensable gases were pumped off through a liquid-nitrogen trap. The volume of the collected gases was measured manometrically, and a sample was taken for mass spectrometric analysis.

The nitrogen-to-hydrogen ratio of noncondensable gas was determined in most of the test units containing more than 5 to 10 cc at standard temperature and pressure (STP). For most of these analyses, the hydrogen content was undetectable. With few exceptions, the hydrogen content of the remaining test units was no more than 4%, and these exceptions were welded or brazed specimens. An unexplainable exception was test unit 4019, which contained a Lee plug, and for which the hydrogen content of the noncondensable gas was 12.5%. The total amount of gas was also high, 4 to 5 times the quantity found with the other three Lee-plug test units.

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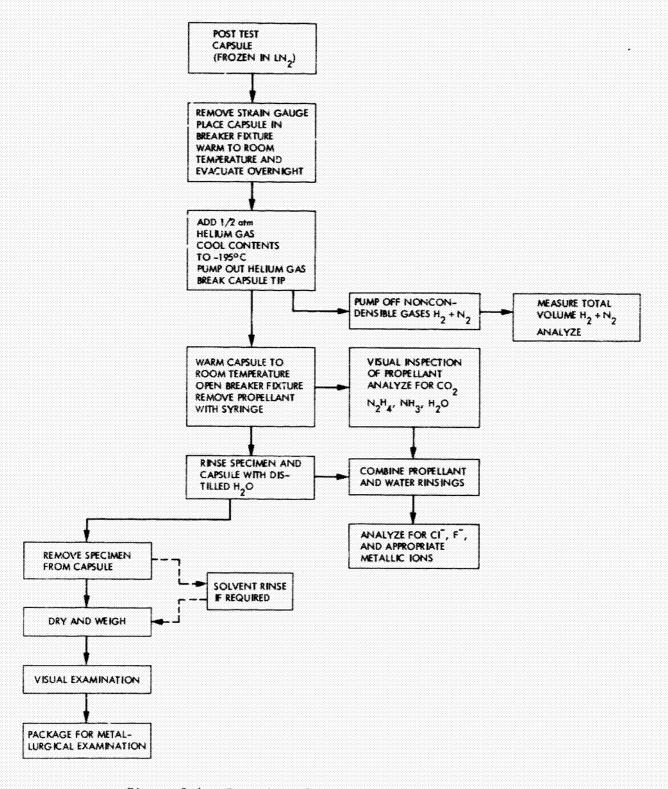


Figure 2-4. Procedure for Posttest Chemical Analysis

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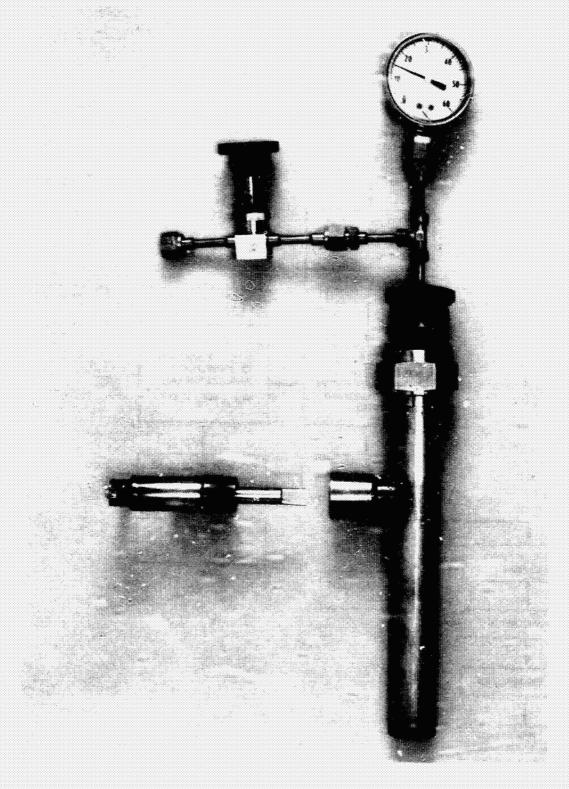


Figure 2-5. Specimen/Capsule Test Opening Fixture

The liquid nitrogen traps were replaced by traps at -30° C (-22° F). After repeated thawing and refreezing of the hydrazine, the remaining condensable gases (mainly NH₃) were pumped off, measured, and sampled. The fixture was opened and the hydrazine removed using a syringe.

b. Calculated Final Capsule Pressure. The mean volume of the test capsules was 82 cm^3 . With 40 g of hydrazine and a standard metal coupon, the ullage was about 40 cm^3 . The pressure calculations depend upon the volume of nitrogen plus hydrogen (assuming negligible solubility in the hydrazine) and the volume of ammonia in the vapor phase.

The contributions of nitrogen and hydrogen were calculated from the perfect gas law:

$$P_{v} = \frac{NRT}{V} \tag{1}$$

where

 P_1 = partial pressure of gas in atmospheres

 \hat{N} = moles of gas = cm³ gas (STP)/22,400

R = universal gas constant = 82.05 cm³-atm/deg-mole

 $T = 316.6 \text{ K} (110^{\circ}\text{F}) \text{ or } 333 \text{ K} (140^{\circ}\text{F})$

V = ullage volume of capsule, cm³.

The calculation of pressure due to the ammonia is not so simple and straightforward. Ammonia is highly soluble in hydrazine, and may not necessarily be an ideal gas at the temperatures and pressures considered.

Fortunately, solubility data for ammonia in hydrazine are available (Reference 5). Although the data do not cover the temperatures of interest, viz., 43°C and 60°C, it was possible to extrapolate the data of the above-referenced report. It can be shown that for the ammonia dissolved in hydrazine, the following relationship can be used to determine a close approximation of the ammonia pressure:

$$P = \frac{N/m}{K} \tag{2}$$

where

P = partial pressure of ammonia, atm

 $N = mc^{1}es$ of ammonia in system

m = moles of hydrazine in system

 $K = \text{equilibrium constant } (0.0455 \text{ atm}^{-1} \text{at } 43^{\circ}\text{C}, 110^{\circ}\text{F}),$ (0.0295 atm⁻¹ at 60°, 140°F)

- c. Pressure Rise Rate. It would be of interest, both practically and theoretically, to have curves expressing pressure as a function of time for each test capsule. Unfortunately, due to aging of the bonding material, some of the strain-gauge data have proven to be unreliable.
- d. Percentage of Hydrazine Decomposed. The percentage of hydrazine decomposed is calculated from the total weight of the gaseous products of decomposition viz, nitrogen and ammonia. Some hydrogen may arise from the attack of metals by acidic constituents, but the error, if any, is insignificant because of the low molecular weight of hydrogen.

5. Residual Hydrazine

The residual hydrazine was removed from the capsule and analyzed as follows:

a. Impurities: NH₃ and H₂O. The NH₃ and H₂O contents of the hydrazine were analyzed by gas chromatography using a 0.0065-m-diam x 2-m-long (1/4-in.-diam x 6-ft-long) column filled with powdered Teflon coated with 15 wt% tri-ethanolamine. The inlet and column temperatures were held at 90° C (194°F) and the helium flow set at 100 cm 3 /min. This column separates NH₃, H₂O, and N₂H₄, in that order.

b. Contaminants: Metals, Halogen and CO2.

- (1) Metals. The capsule was cut open and the metal sample rinsed with water, while any adhering material was rubbed loose with a rubber policeman. Any residue in the capsule was also rinsed out. All washings and residue were acidified with 5% HNO3, diluted to a known volume with water and analyzed for the appropriate metals by atomic absorption.
- (2) <u>Halogens</u>. An aliquot of the acidified washings was checked for chloride by turbidimetry. If the chloride content was high, titration was used. The fluoride ion content was determined by a spectrophotometric method based on the bleaching of a zirconium alizarin color complex by fluoride ion. The absorbance was measured at 525 nm.
- (3) <u>Carbon dioxide</u>. Hydrazine reacts with carbon dioxide to form the salt, hydrazinium carbazate. The equation for this reaction is

$$CO_2 + 2N_2H_4 + (N_2H_5)$$
 (NH₂NHCOO).

The method of analysis involved the addition of a sample of hydrazine to an excess of sulfamic acid. The sulfamic acid liberates CO₂ from the hydrazinium carbazate. Sulfamic acid was selected for use in the analysis because hydrazinium sulfamate is soluble in water and sulfamic acid is nonvolatile.

The liberated CO_2 was swept out of solution with helium gas, through a trap containing concentrated sulfuric acid to remove the water, and then through a special trap containing small glass beads where the CO_2 present was frozen out at the temperature of liquid nitrogen.

The trap containing the frozen CO_2 was provided with a special four-way stopcock that permits the CO_2 to be isolated in its loop. This trap is attached to a special sample introduction system on a custom-built chromatograph that permits the collected CO_2 to be quantitatively transferred through a chromatographic column for separation and assay. (Refer to Appendix C for details of this mathod.)

SECTION III

TEST RESULTS

A. DESCRIPTION OF TEST SPECIMENS

To determine the hydrazine/tank material compatibility, tests were performed on 26 types of metallic and nonmetallic coupons obtained from a fabricated tank, as shown in Figure 1-2. A detailed description of these coupons by CPR numbers and the results of the posttest visual examination are discussed in this section and are compiled in Table 2. Photographs of all test coupons in the posttest condition are presented in Appendix D. Additional data on 347 CRES weld specimens from the JPL/NASA long-term program are presented in Appendix E. A summary of posttest visual examination of coupons is presented below.

- a. $\underline{\text{CPR 1}}$. Specimens 001 to 008 are Type-304L corrosion-resistant steel (CRES) coupons from the ARDE, Inc., diaphragm. All specimens were observed to have a matte finish with no visible corrosion on their surfaces.
- b. <u>CPR 2</u>. Specimens 014 to 017 are Type-304L CRES coupons from the ACS tank liner/diaphragm assemblies, which were girth-welded. All four specimens were shiny in appearance with no surface corrosion apparent. Specimen 014 had a very thin film along the heat-affected zone (HAZ) of the liner. Specimens 015 and 016 also had thin films along the HAZ of the diaphragm.
- c. <u>CPR 3.</u> Specimens 023 to 026 are coupons from burst-disc assemblies made of Type-347 CRES preformed sheet stock, which was electron-beam (EB) welded to a ring made of Type-304L CRES. Each specimen was shiny and bright with no evidence of corrosion on its surface. Each diaphragm had identification numbers and symbols inscribed on it, in addition to the preformed scoring marks. All specimens were observed to have very small parallel scratches on the surfaces.
- d. <u>CPR 4.</u> Specimens 034 to 037 are commercial Lee plugs made of Type-304L CRES. All four specimens had shiny and bright surfaces with no corrosion evident.
- e. <u>CPR 5.</u> Specimens 045 to 048 are coupon sections from the Type-3041. CRES liner/diaphragm liquid outlet housing, which was tungsten-inert-gas (TIG) welded. All specimens appeared bright and shiny with no signs of surface corrosion apparent.
- f. <u>CPR 6</u>. Specimen^c '56 to 059 are coupon sections from the Type-304L CRES EB weld joint No. 407. All specimens' metallic surfaces were shiny and bright with no signs of visible corrosion.
- g. $\underline{\text{CPR 7}}$. Specimens 067 to 070 are coupon sections from the Type-304L CRES EB weld joint No. 406. All coupons' metallic surfaces were shiny and bright with no signs of visible corrosion.

- h. <u>CPR 8.</u> Specimens 078 to 081 are coupon sections of Types 308L and 304L CRES from the stiffening ring/diaphragm spot-welded subassemblies. All were shiny and bright with no evidence of surface corrosion.
- i. <u>CPR 9.</u> Specimens 300 to 307 are coupon sections of Type-304L CRES from the ACS tank liner. All samples were shiny, with a matte finish. No corrosion was noted on these coupons.
- j. <u>CPR 10</u>. Specimens 100 to 103 are coupon sections from the tank shell composed of Type-A286 CRES. The metal was still shiny with a minor amount of gray discoloration. No corrosion on the coupon was evident at 45x magnification.
- k. CPR 11. Specimens 109 and 110 are coupon sections from TIG-welded joint No. 41 of Type-A286 CRES, which was a part of the ACS tank girth weld. The metal was shiny except for a gray discoloration in the HAZ along each side of the weld. No corrosion was evident.
- 1. CPR 12. Specimens 112 and 113 are coupon sections of the TIG-welded joint No. 417 composed of Type-A286 CRES, which was welded to Type-304L CRES. The specimen metal surfaces were shiny and bright with no evidence of corrosion.
- m. $\overline{\text{CPR }13}$. Specimens 115 and 116 are sections of Type-308L CRES filler wire used in the diaphragm stiffening ring. The wire was shiny except for dark-gray bands about 1/2 in. from each end. No other corrosion was evident.
- n. <u>CPR 14</u>. Specimens 118 to 121 are coupon sections of Type-304L CRES from the liner-diaphragm assembly with a gold-nickel brazed Type-308L CRES filler wire. All coupons were shiny and bright with no evidence of corrosion.
- o. <u>CPR 15.</u> Specimens 127 and 128 are pieces of the Mylar plastic sensor disc from the vapor-detection assembly. Both specimens dissolved in the hydrazine propellant after three hours of testing. (The nydrazine became light yellow in color and contained 0.016 mg of iron.)
- p. <u>CPR 16.</u> Specimens 130 and 131 are coupon sections of Type-304L CRES from the liquid outlet housing to the tank-half EB-welded joint No. 404. Specimen 130 was shiny except for some etching on the bottom of the coupon and a gray discoloration in the HAZ along each side of the weld. Specimen 131's surface was shiny and bright except for an irregular surface on the top. No corrosion was seen on either coupon.
- q. <u>CPR 17</u>. Specimens 133 and 134 are fluorinated-ethylenepropylene-(Teflon-) coated samarium-cobalt magnets from the vapor-detection assembly. The Specimen 153 coating was intact, but uneven. No corrosion was evident. The posttest weight was significantly lower than the pretest weight. A reason for the loss was not readily apparent. However, it was observed that the magnet did influence the action of the scale balance; an accurate weight was obtained only after neutralizing the magnetic influence. The specimen-134 Teflon coating was irregular but apparently intact. There were dark spots on the magnet surface, but it was not possible to identify them as corrosion spots.

- r. $\underline{\text{CPR }18}$. Specimens 200 and 201 are coupon sections of Type-17-4 PH CRES, H 1050 temper, and electropolished, from the piston in the flow-equalizer valve. The surface of the specimens was bright with no evidence of corrosion.
- s. $\overline{\text{CPR 19}}$. Specimens 210 and 211 are electropolished and chrome-plated sections of Type-17-4 PH CRES, H 1050 temper, from the shaft end of the flow-equalizer valve. The specimens were shiny and bright with no corrosion evident.
- t. <u>CPR 20</u>. Specimens 220 and 221 are springs of electropolished Type-17-4 PH CRES, CH 900 temper, from the flow-equalizer valve. Specimen 220 is bright with a light-gray tarnish. No other corrosion was evident. Specimen 221 was shiny with no corrosion evident.
- u. <u>CPR 21</u>. Specimens 230 and 231 are coupon sections of Type-17-4 PH CRES, TIG welded and electroplated, from the shaft end assembly of the flow-equalizer valve. The metal was shiny and bright with some etching near the coupon identifying number. No corrosion was seen.
- v. <u>CPR 22</u>. Specimens 240 and 241 are bourdon tubes of Inconel 902 (Ni Span C) from the pressure-switch assembly. The metal surfaces have an even gray oxidized coating with no corrosion evident.
- w. <u>CPR 23</u>. Specimens 250 and 251 are coupon sections and pieces of Type-347 CRES bar stock from the propellant distribution manifold fitting. Specimen 250 was shiny with a very light-gray mottling perceptible on the surfaces. No corrosion was apparent. Specimen 251, with circular machining marks, was shiny and bright. No corrosion was seen.
- x. <u>CPR 24</u>. Specimens 260 and 261 are of Type-347 CRES tubing. A light-gray tarnish was evident on the surface, but no corrosion can be seen.
- y. <u>CPR 25</u>. Specimens 270 and 271 are coupon sections of Type-347 CRES tubing Astroarc welded (Weld No. 78) to Type-347 CRES tubing. The tubing was shiny except for a gray discoloration in the HAZ on each side of the weld. No corrosion was evident.
- z. <u>CPR 26</u>. Specimens 280 and 281 are O-rings made of Parker seal compound EPR 515. Microscopic examination of the O-rings revealed no crazing or cracking; the surface appeared smooth and unbroken. There was no Krytox 240AC coating on the specimens before cleaning.

B. DETAILS AND SUMMARIES OF POSTTEST ANALYSES AND RESULTS

The posttest analyses and results are summarized in Table 2. The duration of the test units in storage, test temperatures in degrees Celsius, and capsule posttest pressures at test temperature in $\rm N/cm^2$ are given. The specimen material, configuration, and weight change in milligrams are listed. The specimen age decomposition of hydrazine and the gas evolution rate in cm 3 x 10^{-3} day 1 . cm $^-$ are also given.

Data on the individual test units, test specimens, and the hydrazine propeliant is given in Table 3. The BAT number is identified with the test unit number. The initial weight in grams and the change in weight is given for each specimen. The analysis of the hydrazine is given in milligrams for dissolved iron (Fe) and by percent for water (H₂O) and ammonia (NH₃). The hydrazine decomposition into the noncondensable gases nitrogen (N₂) and hydrogen (H₂) is given as total volume (cm³) at standard temperature and pressure (STP); the gas evolution rate, both uncorrected and control corrected, is in cm³ x 10^{-3} . day 1^{-2} . cm⁻².

C. PROPELLANT CONTROLS

Table 4 presents data on the hydrazine propellant, unit number, days on test, temperature in degrees Celsius, capsule pressure at test temperature, and the $\rm H_2O$ and NH₃ analyses, by percent, along with information on hydrazine decomposition. The noncondensable gases (N₂ and H₂) are listed as total volume cm³ at STP and the rate as cm³ x 10⁻³ . day⁻¹.

D. SURFACE ANALYSIS

1. Introduction

As noted in Table 2, in a few pairs (or groups) of specimens, one of the test units shows a significantly higher gas evolution than the others in that group. For example, specimen BA 008 had twice the gas evolution rate of any of the others in the CPR I group. In the CPR 4 group, one of the Lee plugs greatly enhanced the decomposition of hydrazine. Other examples can be seen in Table 2.

Two sets of specimens were chosen for very scrupulous examination by X-ray photoelectron spectroscopy (XPS) and the scanning electron microscope (SEM). Both sets are from the primary containment system (Program B) and both of 304L CRES. The specimens examined were BA 005 and BA 008 of the CPR 1 p, and BA 305 and BA 307 of the CPR 9 group. In each case an untreated cimen, i.e., one not exposed to hydra ine, was used as a control in the analysis.

2. XPS Techniques

The six specified stainless-steel samples were submitted for surface analysis by the XPS technique; Figure 3-1 shows the JPL XPS laboratory. The samples were cut from the original strips using metal shears. Then, immediately prior to insertion into the XPS spectrometer, each sample was cleaned ultrasonically in absolute ethanol for 10 minutes and dried with flowing nitrogen. The following samples were analyzed:

| Set 1 | Set II |
|-----------------|-----------------|
| CPR I (CONTROL) | GPR 9 (CONTROL) |
| BA 005 | BA 305 |
| BA 008 | BA 307 |

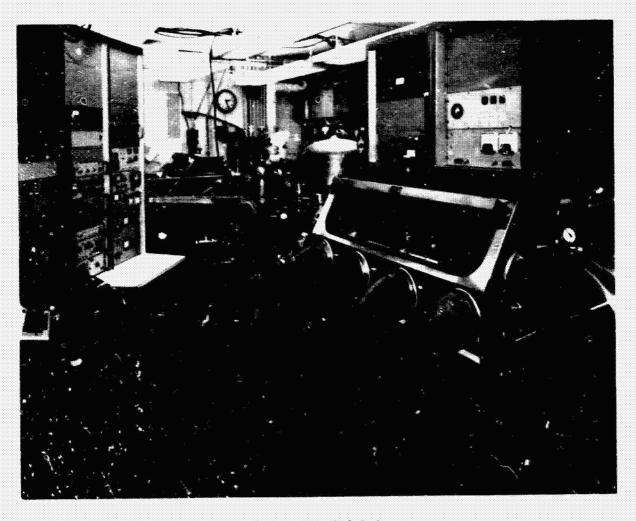


Figure 3-1. JPL XPS Laboratory

Analyses were performed with a modified Hewlett-Packard 5950A electron spectrometer under computer control. Photoemicted electrons, characteristic of the surface composition of the sample, were formed by interaction with 1486.6-eV incident photons from an aluminum K α radiation source. For the instrumentation employed, the measured photoemission represents an average signal over an area of approximately 1 mm x 5 mm. Although the photoemitted electrons from a given atomic core level may sometimes originate as much as 100 Å below the sample surface, the immediate surface region of the sample will actually contribute significantly more to the measured signal due to an exponential attenuation (with depth) of photoelectrons by the solid.

For each sample, a wide energy-range (100 to 1300 eV) scan was run to determine the major contributors to the total photoelectron spectrum, followed by careful measurement of a 20 to 40 eV binding energy-range characteristic of each of eight elements of major interest. Each such run for a given sample took a total of 15.2 hours of instrument time to obtain adequate statistics. Photoelectron spectra (PES) of the following elements were obtained:

| Element | Energy <u>Levels</u> | Measured Binding Energy Range, eV |
|---------|---|--------------------------------------|
| С | ls | 275 to 295 |
| N | ls | 390 to 410 |
| 0 | ls | 523 ro 543 |
| Cr | 2p3/2, 2p1/2 | 560 to 600 |
| An | ² P ₃ / ₂ , ² P ₁ / ₂ | 625 t a 665 |
| Fe | ² P3/2, ² P1/2 | 695 to 735 |
| Ni | ² P ₃ / ₂ , ² P ₁ / ₂ | 845 to 895 |
| Zn | ² p _{3/2} , ² p _{1/2} | 1010 to 1050 |

The XPS BE (binding energy) spectra can be used to identify different elements on a surface and to distinguish between the same element in different chemical environments. For example, exidation of a metal causes an effective unbalanced positive change localized on the metal atom and the remaining electrons are therefore held more tightly; i.e., their binding energy increases.

3. Results

For the various spectral regions examined in this investigation, some general observations are presented below.

a. <u>Carbon Is Region.</u> Carbon is the major constituent of the surface of all samples, amounting to 40 +4 atomic percent (at.%) for five samples and to a significantly higher 68.8 at.% for CPR I (which also gave anomalous results in almost all the other measurements). The primary peak in the carbon spectra comes from aliphatic carbon compounds, while there are also substantial intensities in the regions expected for carbon-nitrogen compounds (~286 eV) and carbon-oxygen compounds (~288 eV). A quantitative measure of the relative amounts of these three carbon species may be obtained from the application of computer curve-fitting routines to the individual spectra.

- b. Oxygen is Region. The apparent surface concentration of oxygen is approximately the same (46 +3 at.%) for all samples, with the exception of CPR 1(24.7%). In the case of oxygen, a variety of metal oxides and hydroxides contribute to the measured signals and, in the absence of adequate reference data, it is very difficult to make any specific assignments to the various peaks and shoulders observed. It should be noted that the oxygen is binding energy for CPR i is shifted to a higher energy than that observed for the other five samples, suggesting a significantly different distribution of metal oxides/hydroxides for this particular sample, when compared to the others.
- c. Nitrogen Is Region. The apparent surface concentration of nitrogen is observed to be approximately the same (2.8 ±0.5 at.2) for all six samples. The measured binding energy of the nitrogen peak is, for all samples, consistent with that to be expected for protonated amines and amino polymers.
- d. Chromium 2p Region. In both Sets I and II, the apparent surface concentration of chromium for the samples exposed of the control specimens, CPR 9 (4.3 at.%) or CPR 1 (6.8 at.%, anomalously low). The predominant species present in all samples are metallic chromium (relatively small) and chromium oxide or hydroxide (relatively large). The ratio of metallic to oxidized chromium is approximately the same for all samples.
- e. Manganese 2p Region. In both Sets I and II, the apparent surface concentration of manganese for the samples exposed to hydrazine (1.0 +0.2 at.%) is found to be greater than that for either of the control specimens CPR 9 (0.5 at.%) or CPR 1 (0.1 at.%, again low). The predominant species present in all samples is oxidized manganese as the monoxide; hydroxides may be present as well, due to the close similarity of binding energies.
- f. Iron 2p Region. The dominant surface species present in all samples are metallic iron and ferric oxide; iron hydroxides are also possible since oxides and hydroxides exhibit similar binding energies. In both Sets I and II, the ratio of metallic iron to oxidized iron is much greater for the samples subjected to hydrazine than for the control specimens. In Set II, the control specimen CPR 9 has a greater total iron surface concentration (3.9 at.% than that for the samples contacted b, hydrazine (3.1 +0.8 at.%). In Set I just the opposite occurs; the total surface iron for CPR 1 (1.0 at.%) appears to be anomalously low.
- g. Nickel 2p Region. In all cases, the primary peak in the nickel spectrum is due to metallic nickel; a much less intense peak at higher binding energy is due to the monoxide. In both Sets I and II, the apparent surface concentration of nickel was approximately the same (0.3 at.%) for the samples exposed to hydrazine. The nickel concentration for control sample CPR 9 was somewhat lower (0.1 at.%), while for CPR 1 it was exceptionally low (0.2 at.%).
- h. Zinc 2p Region. In all cases, the surface zinc is present in oxidized form, probably as the simple oxide. In Set I, the apparent surface concentration of zinc is approximately equal (0.2 at.%) for all three samples. In Set II, the control specimen, CPR 9, shows 2 to 3 times more zinc (0.6 at.%) than the samples subjected to hydrazine.

4. Discussion

Both the carbon and nitrogen binding energy spectra give strong evidence of surface contamination by carbon-nitrogen compounds such as amino polymers. It should be noted that although these samples were apparently stored in polyethylene bags in pretreatment steps, the <u>final</u> pretest step consisted of heat-sealing in nylon, a process which could readily provide the observed amino polymers on the surface.

The anomalous results for control CPR 1 were confirmed by a second run which gave results identical to the original run. This specimen was more reduced in oxygen and metals concentration, but more heavily contaminated by carbon than any of the other five samples. Therefore, more meaningful comparison may probably be made using CPR 9 as a control for both sets of samples subjected to hydrazine.

The overall picture is that, upon exposure to hydrazine, oxidized iron is removed, leaving a surface richer in the protective chromium oxide. A more thoughtful analysis may be aided by taking into account the information presented in Reference 6, and the references contained therein.

5. Scanning Electron Microscopy (SEM) Examination

These same specimens were then examined by SEM to look for differences in surface morphology. The surfaces of specimens BA 005 and BA 008 were identical in appearance when examined at 50x and 500x magnifications. When compared to the CPR 1 control, the specimens exposed to hydrazine appeared to have very minor surface pitting. The surfaces of specimens BA 305 and BA 307 also were identical and no differences could be seen when compared to the CPR 9 control.

In addition, the specimen BA 036, Lee plug, was thoroughly examined by SEM because of the greater decomposition rate of its test unit compared to the three other replicates. A direct comparison to specimen control BA 038 indicated no differences.

6. Conclusions

These highly detailed and sensitive analyses failed to indicate the causes for differences observed in the rates of hydrazine decomposition between pairs of nominally identical specimens. Some of the more subtle differences in surface character were unfortunately masked by the presence of significant contamination by carbon-nitrogen compounds from the sealed nylon storage bags. The thickness of this carbon-nitrogen layer is such (<100Å) that it would have no effect on either the rate of hydrazine decomposition or the effect of corrosion of the coupons. SEM examination of the above-mentioned six coupons and Lee plug BA 036 again revealed no cause for the difference in decomposition rates.

SECTION IV

CONCLUSIONS

With few exceptions, mainly attributable to catalysis, possible contamination, or inherent sample-to-sample variation, the rate of hydrazine decomposition in these tests was very low -- producing less than 1.0 cc of gas per year per cm² of specimen area.

The degree of corrosion of the metal coupons was virtually unmeasurable in all instances. The elastomer EPR-515 did not appear to degrade, and the Mylar film dissolved as expected.

A. PROGRAM A: 6 MONTHS STORAGE

The rates of hydrazine decomposition were low in most test units — less than $1.0~\rm cm^3$ gas per year per cm² of specimen area. The following were a few exceptions to the low rates:

- 304L liner-diaphragm with Au-Ni brazed 308L filler wire (BA 118-121), 1.0 to 3.7 cm³/yr/cm²
 - (a) Possible catalysis by Au-Ni braze
 - (b) Rate based on total coupon area.
- (2) FEP-coated Sm-Co magnets (BA 133-134), 3.7 to $13.2 \text{ cm}^3/\text{yr/cm}^2$
 - (a) FEP coating intact
 - (b) Possible permeation and catalysis.
- (3) ERP-515 O-rings
 - (a) Catalysis by carbon black used in compounding elastomer
 - (b) Very rapid decomposition, but area rate not meaningful.
- (4) Mylar film (BA 127-128), $3.5 \text{ cm}^3/\text{yr/cm}^2$
 - (a) Film dissolved
 - (b) Area rate not meaningful.

The corrosion of metallic coupons was minimal and only very light tarnish was seen on a few specimens. The weight changes of coupons were negligible, and dissolved iron in the propellant was almost unmeasurable. Two nonmetals were included in Program A:

- (1) Mylar film (BA 127-128), which dissolved as expected.
- (2) EPR-515 O-rings (BA 280-281), which appeared unchanged after exposure to hydrazine.

B. PROGRAM B: 24 MONTHS STORAGE

The rates of hydrazine decomposition were low in most test units — less than 1.0 cm³ gas per year per cm² of specimen area. There were a few exceptions to low rates, but the results were not consistent:

- (1) 304L liner/diaphragm girth weld (BA 017), 1.3 cm³/yr/cm²
 - (a) Only one of four specimens produced an anomalously large volume of gas
 - (b) Possible contamination or sample-to-sample variation.
- (2) 304L Lee plug (BA 036), $5.2 \text{ cm}^3/\text{yr/cm}^2$
 - (a) Only one of four specimens showed a high rate of decomposition
 - (b) Possible contamination or sample-to-sample variation.
- (3) 304L EB weld No. 407 (BA 058-BA 059), $2.3-3.3 \text{ cm}^3/\text{yr/cm}^2$
 - (a) Two specimens at 43°C showed low rate of gas formation
 - (b) Possible effect of 60°C storage
 - (c) Possible contamination or sample-to-sample variation.
- (4) 304L EB weld No. 406 (BA 070), $1.8 \text{ cm}^3/\text{yr/cm}^2$
 - (a) Only one of two at 60° C was high
 - (b) Possible contamination or sample-to-sample variation.

None of the specimens appeared to corrode, and only a very light tarnish was seen on a few specimens. Weight changes of the coupons were negligible, and the dissolved iron in the propellant was almost unmeasurable.

In general, the results of this study agree very well with the JPL/NASA long-term compatibility program (References 2 and 3). The Type-304L CRES chosen for the primary containment of hydrazine in the propellant tank appears to be entirely suitable for use in systems requiring at least a 2-year service life. In the secondary containment side of the tank, the A286 CRES used in the outer pressure vessel is compatible with hydrazine for 6 to 12 months exposure. The Au-Ni braze material, FEP-coated Sm-Co magnets, and the EPR 515 O-rings have been shown to cause hydrazine decomposition that could result in an undesirable gas pressure buildup which must be accomposited in the system design. However, these materials would be in contact with hydrazine only if a leak occurred in the primary containment system.

SECTION V

DATA TABLES

The tables are, generally, self-explanatory. The following comments are given to expand on certain topics.

The strain gauge data shown in Table 2 indicate that this is not a reliable method of determining capsule pressures of less than one atmosphere. The strain gauges are normally calibrated at positive pressure only, and an extrapolation is made to zero pressure. Attempts to calibrate a capsule-mounted strain gauge at subatmospheric pressure produced results that indicated random shifting of the calibration line. At pressures greater than one atmosphere, the strain gauge data agree very well with the actual pressures found in the capsules. Posttest recalibration of several capsules indicates that while the zero point may shift, the sensitivity is maintained during handling and testing.

The decomposition of hydrazine in the control capsules (Table 4) presumably occurs through homogeneous (bulk) catalysis; glass should not act as an active surface for hydrazine decomposition. Purified hydrazine contains very little dissolved iron (a known catalyst) and, therefore, the rate of decomposition is predictably slow. With the introduction of a metallic specimen, there is the possibility of an active surface and heterogeneous (surface) catalysis. If metal is dissolved from the surface of the specimen, it is possible for both reaction mechanisms to occur. Obviously, from an inspection of some of the results (Tables 2 and 3), there are metallic surfaces which are not catalytically active towards hydrazine, especially Type-304L CRES Alloy.

OF POOR QUALITY

Table 1. Listing of Coupon Test Numbers and Description

| | Test Numbers | | Manual Companibility (Ban Complete Broadester |
|-----|--------------|-----------|--|
| CPR | BAT | Test Unit | Material Compatibility Test Specimen Description |
| 1 | 001-008 | 4001-4008 | 304L Arde diaphragm |
| 2 | 014-J17 | 4009-4012 | 304L/304L liner/diaphragm girth weld |
| 3 | 023-026 | 4013-4016 | 347 burst disc |
| 4 | 034-037 | 4017-4020 | 304L Lee plug |
| 5 | 045-048 | 4021-4024 | 304L/304L iiner/diaphragm outlet housing TIG weld |
| 6 | 056-059 | 4025-4028 | 304L/304L EB weld #407 |
| 7 | 067-070 | 4029-4032 | 304L/304L EB weld #406 |
| 8 | 078-081 | 4033-4036 | 308L/304L ring/diaphragm spot welded |
| 9 | 300-307 | 4037-4044 | 304L liner |
| 10 | 100-103 | 4045-4048 | A286 tank shell |
| 11 | 109-110 | 4049-4050 | A286/A286 girth TIG weld #411 |
| 12 | 112-113 | 4051-4052 | 304L/A286 polar TIG weld #417 |
| 13 | 115-116 | 4053-4054 | 308L stiffening ring |
| 14 | 118-121 | 4055-4058 | 308L/304L wire/diaphragm, Au-Ni braze |
| 15 | 127-128 | 4059-4060 | Mylar sensor disc |
| 16 | 130-131 | 4061-4062 | 304L/304L EB weld #404, liquid outlet housing to tank half |
| 17 | 133-134 | 4063-4064 | Samarium-cobalt magnet, FEP coated |
| 18 | 200-201 | 4065-4066 | 17-4 PH, H 1050 temper, electropolished |
| 19 | 210-211 | 4067-4068 | 17-4 PH, H 1050 temper, electropolished, chrome plated |
| 20 | 220-221 | 4069-4070 | 17-4 PH, CH900 temper, spring |
| 21 | 230-231 | 4071-4072 | 17-4 PH/17-4 PH, TIG weld, shaft end |
| 22 | 240-241 | 4073-4074 | Inconel 902, Ni span C, Bourdon tube |
| 23 | 250-251 | 4075-4076 | 347 manifold fitting |
| 24 | 260-261 | 4077-4078 | 347 tube, annealed |
| 25 | 270-271 | 4079-4080 | 347/347 Astro-arc weld #78 |
| 26 | 280-281 | 4081-4082 | EPR 515, Parker seal, O-ring, Krytox coated |
| | | | |

Table 2. Summary of Analyses and Results

| BAT No. | Days Test on Unit Test | | | | | | | Specimen | | F | ropellant | | | | |
|------------|------------------------------|-----|----|-------|--------|-----------|------------------|----------|------|----------------------|----------------------|---|-----------------------------|----------|---------------|
| | | | | | | | | | | on | Test Temp., OC | Capsule Pressure at Test Temp., N/cm ² | (Strain Gage Reading) | Material | Configuration |
| BA001 | 4001 | 807 | 43 | 0.77 | (0) | 304L CRES | ACS Diaphragm | -0.4 | 0.02 | 0.01 | | | | | |
| BA002 | 4002 | 807 | 43 | 0.72 | (0) | •• | 19 | 0.0 | 0.02 | -0.01 | | | | | |
| BA003 | 4003 | 807 | 43 | 0.99 | (0) | ** | ** | -0.2 | 0.03 | 0.06 | | | | | |
| BA004 | 4004 | 807 | 43 | 0.65 | (0) | ** | •• | -0.1 | 0.01 | -0.01 | | | | | |
| BA005 | 4005 | 765 | 60 | 1.70 | (0) | ** | " | +2.5 | 0.02 | -0.10 | | | | | |
| BA006 | 4006 | 765 | 60 | 2.85 | (0) | ** | 19 | -0.2 | 0.05 | 0.10 | | | | | |
| BA007 | 4007 | 765 | 60 | 2.12 | (0) | •• | ** | 0.0 | 0.04 | -0.03 | | | | | |
| BA008 | 4008 | 765 | 60 | 3.10 | (0) | •• | ,, | -0.3 | 0.06 | 0.19 | | | | | |
| BA014 | 4009 | 807 | 43 | 0.89 | (0) | ** | Liner/diaphragm, | -0.8 | 0.02 | 0.02 | | | | | |
| BAu15 | 4010 | 807 | 43 | 1.41 | (0) | ** | Girth weld | -1.2 | 0.02 | 0.10 | | | | | |
| BA016 | 4011 | 807 | 60 | 3.25 | (0) | ** | ** | -0.9 | 0.06 | 0.10 | | | | | |
| BA017 | 4012 | 765 | 60 | 33.39 | (35.1) | ** | ** | -0.5 | 0.97 | | | | | | |
| BA023 | 4013 | 378 | 43 | 2.07 | (5.0) | CRES 347 | Burst Disc | -0.4 | 0.12 | 0.74 | | | | | |
| BA024 | 4014 | 378 | 43 | 2.48 | (0) | ** | •• | -0.4 | 0.16 | 0.74 0.95 | | | | | |
| BA025 | 4015 | 378 | 60 | 9.81 | (0) | ** | 11 | -0.4 | 0.41 | 3.91 | | | | | |
| BA026 | 4016 | 378 | 60 | 8.24 | (0) | ,, | •• | -0.1 | 0.35 | 3.14 | | | | | |
| BA034 | 4017 | 835 | 43 | 1.05 | (0) | 304L CRES | Lee Plug | -0.4 | 0.03 | 3.14 0.61 2.40 | | | | | |
| BA035 | 4018 | 835 | 43 | 1.95 | (0) | •• | " | -0.3 | 0.04 | 2.40 | | | | | |
| BA036 | 4019 | 835 | 60 | 10.98 | (5. | ** | 11 | +15.5 | 0.26 | 14.10 | | | | | |
| BA037 | 4020 | 835 | 60 | 3.77 | (0) | •• | •• | +7.1 | 0.04 | 2.62 | | | | | |
| BA045 | 4021 | 835 | 43 | 1.12 | (8.0) | ** | TIG weld | +0,1 | 0.03 | 6.29 | | | | | |
| BA046 | 4022 | 835 | 43 | 1.30 | (0) | •• | ** | -0.3 | 0.04 | 0.33 | | | | | |
| BA047 | 4023 | 835 | 60 | 2.80 | (0) | ** | ** | -0.2 | 0.06 | 0.28 | | | | | |
| BA048 | 4024 | 835 | 60 | 7.44 | (2.5) | ** | " | 0.0 | 0.14 | 2.90 | | | | | |
| BA056 | 4025 | 771 | 43 | 2.73 | (0) | ** | EB weld #407 | -1.0 | 0.09 | 0.44 | | | | | |
| BA057 | 4026 | 771 | 43 | 6.81 | (2.1) | •• | ** | -1.0 | 0.25 | 1.18 | | | | | |
| BA058 | 4027 | 729 | 60 | 49.35 | (38.6) | •• | ** | -1.1 | 1.40 | 9.00 | | | | | |
| BA059 | 4028 | 715 | 60 | 35.34 | (29.6) | ** | n | -1,2 | 1.09 | 6.22 | | | | | |

Table 2. Summary of Analyses and Results (continuation 1)

| | | | | | | | Specimen | | Pr | ropellant | |
|------------|--------------|--------------------|----------------------------------|---|-----------------------------|----------------|------------------|------------------------|----------------|--|---------------------|
| BAT No. | Test Unit | Days on Test | Test Temp., ^O C | Capsule Pressure at Test Temp., N/cm ² | (Strain Gage Reading) | Material | Configuration | Weight Change mg | Decomposition, | Gas Evolution cc × 10 ⁻³ • day • cm ⁻² | -1 |
| BA067 | 4029 | 771 | 43 | 1.27 | (0) | 304L CRES | EB weld #406 | -0.7 | 0.02 | 0.14 | |
| BA068 | 4030, | 771 | 43 | 2.97 | (3.4) | ** | 11 | -0.8 | 0.10 | 0.49 | |
| BA069 | 4031 | 757 | 60 | 8.10 | (0) | •• | ** | +142.28 | 0.28 | 1.28 | |
| BA070 | 4032 | 715 | 60 | 25.15 | (16.5) | ** | ** | -0.8 | 0.77 | 4.80 | |
| BA078 | 4033 | 799 | 43 | 1.05 | (0) | 308L/304L CRES | Ring/diaphragm, | -0.4 | 0.03 | 0.05 | |
| BA079 | 4034 | 799 | 43 | 0.85 | (0) | ** | Spot weld | 0.0 | 0.02 | 6.02 | |
| BA080 | 4035 | 785 | 60 | 6.17 | (0) | •• | " | 0.0 | 0.16 | 0.57 | |
| BA081 | 4036 | 785 | 60 | 9.37 | (1.8) | ** | • | 0.0 | 0.26 | 1.07 | |
| BA300 | 4037 | 807 | 43 | 0.85 | (0) | 304L CRES | Liner | -0.9 | 0.02 | 0.03 | |
| BA301 | 4038 | 765 | 43 | b | (14.0) | ** | *** | +0.5 | _ | _ | |
| BA302 | 4039 | 807 | 43 | 0.70 | (0) | ** | 11 | -0.6 | 0.01 | < 0.01 | 200 |
| BA303 | 4040 | 807 | 43 | 0.74 | (0) | ** | 11 | -1.2 | 0.02 | < 0.01 | <u></u> |
| BA304 | 4041 | 765 | 60 | c | (0) | ** | 11 | -0.7 | - | - | ORIGINAL OF POOR |
| BA305 | 4042 | 765 | 60 | 3.81 | (0) | ** | 11 | +0.6 | 0.08 | 0.41 | 유무 |
| BA306 | 4043 | 765 | 60 | 3.59 | (0) | •• | 11 | 0.0 | 0.08 | 0.31 | |
| BA307 | 4044 | 765 | 60 | 2.50 | (0) | ** | 11 | -0.5 | 0.04 | 0.08 | PAGE |
| BA100 | 4045 | 184 | 43 | u.69 | (0) | A286 | Tank Shell | -0.7 | < 0.05 | 0.13 | 产品 |
| BA101 | 4046 | 245 | 43 | 2.02 | (0) | ** | 11 | -0.3 | 0.15 | 0.68 | PAGE IS |
| BA102 | 4047 | 308 | 43 | 1.21 | (0) | •• | 11 | -0.9 | 0.10 | 0.14 | |
| BA103 | 4048 | 365 | 43 | 1.02 | (0) | ** | 11 | +0.2 | 0.04 | 0.14 | |
| BA109 | 4049 | 245 | 43 | 1.43 | (2.1) | ** | TIG weld #411 | -0.7 | 0.19 | 2.31 | |
| BA110 | 4050 | 365 | 43 | 1.74 | (1.0) | •• | ** | -0.3 | 0.07 | 0.41 | |
| BA112 | 4051 | 245 | 43 | 3.04 | (2.8) | A286/304L | TIG weld #417 | -0.9 | 0.03 | 3.67 | |
| BA113 | 4052 | 365 | 43 | 3.57 | (1.5) | •• | • | -0.4 | 0.13 | 2.60 | |
| BA115 | 4053 | 245 | 43 | 1.31 | (1.7) | 30 8L | Wire | -0.7 | 0.01 | 2.07 | |
| BA116 | 4054 | 365 | 43 | 0.99 | (0) | ** | 11 | +0.1 | 0.08 | 0.14 | |
| BA118 | 4055 | 220 | 43 | 7.48 | (11.6) | 308L/304L | Liner/diaphragm, | -1.1 | 0.36 | 3.46d | |
| BA119 | 4056 | 281 | 43 | 11.39 | (10.0) | ** | Au-Ni Braze | -0.3 | 0.40 | 5.05d | |
| BA120 | 4057 | 344 | 43 | 10.53 | (10.7) | ** | •• | -0.8 | 0.29 | 3.63d | |

Table 2. Summary of Analyses and Results (continuation 2)

| | | | | | | | Specimen | | Propellant | | |
|------------|--------------|--------------------|----------------------|---|-----------------------------|-----------------|----------------------|------------------------|--------------------------|--|--------------|
| BAT No. | Test Unit | Days on Test | Test Temp., oC | Capsule Pressure at Test Temp., N/cm ² | (Strain Gage Reading) | Material | Configuration | Weight Change mg | Decompo- sition, % | Gas Evolution cc x 10 ⁻³ · day ⁻¹ · cm ⁻² | 1 |
| BA121 | 4058 | 401 | 43 | 29.04 | (29.1) | 308L/304L | Au-Ni Braze | -0.2 | 0.70 | 9.9 8 d | |
| BA127 | 4059 | 365 | 43 | 2.70 | (2.4) | Mylar Film | (Specimen Dissolved) | _ | 0.16 | 9.15 ^e | |
| BA128 | 4060 | 365 | 43 | 2.44 | (1.4) | ** | | - | 0.09 | 9.43° | |
| BA130 | 4061 | 308 | 43 | 1.61 | (0) | 304L | EB weld #404 | -1.1 | 0.01 | 0.53 | |
| BA131 | 4062 | 340 | 43 | 0.97 | (0) | ** | 11 | -1.3 | 0.08 | 0.05 | |
| BA133 | 4063 | 308 | 43 | 4.67 | (0) | Samarium-Cobalt | Magnet, FEP- | -28.7 | 0.11 | 36.17 | |
| BA134 | 4064 | 365 | 43 | 2.25 | (0) | ** | Coated | +0.8 | 0.11 | 10.46 | ~ ~ |
| SE200 | 4065 | 308 | 43 | 2.10 | (0) | 17-4 PH, H1050 | Valve, Electro- | -0.5 | 0.02 | 0.90 | 3.5 |
| SE201 | 4066 | 365 | 43 | 0.92 | (0) | ** | Polished | +0.1 | 0.04 | 0.08 | y 🕯 |
| SE210 | 4067 | 365 | 43 | 0.88 | (3.4) | ** | Valve, E.P., | 0.0 | 0.05 | 0.04 | BOOK BOOK |
| SE211 | 4068 | 184 | 43 | 0.58 | (0) | ** | Chrome plated | -1.1 | < 0.05 | | |
| SE220 | 4069 | 184 | 43 | 0.81 | (6.9) | 17-4 PH, CH900 | Spring | -2.4 | < 0.05 | 0.26 | OUALITY. |
| SE221 | 4070 | 365 | 43 | 1.05 | (5.5) | •• | •• | +0.6 | 0.05 | 0.16 | ΣÉ |
| SE230 | 4071 | 308 | 43 | 0.56 | (0) | 17-4 PH | Valve, TIG weld | -0.4 | | 0.01 | 5' |
| SE231 | 4072 | 365 | 43 | 0.76 | (0) | ** | •• | -0.4 | 0.04 | 0.01 | 7 5 |
| SC240 | 4073 | 308 | 43 | 1.29 | (0) | Inconel 902 | Bourdon tube | -0.3 | 0.01 | 0.11 | |
| SC241 | 4074 | 365 | 43 | 1.71 | (0) | ** | •1 | -0.8 | 0.08 | 0.10 | |
| BA250 | 4075 | 245 | 43 | 1.04 | (1.7) | 347 | Bar Stock | -1.2 | 0.06 | 0.18 | |
| BA251 | 4076 | 365 | 43 | 1.52 | (0) | ** | •• | -0.2 | 0.06 | 0.35 | |
| BA260 | 4077 | 245 | 43 | 0.76 | (4.5) | ** | Tube, annealed | -0.8 | < 0.01 | 0.16 | |
| BA261 | 4078 | 365 | 43 | 0.81 | (3.4) | ** | ** | -0.2 | 0.04 | 0.04 | |
| BA270 | 4079 | 245 | 43 | 1.97 | (3.8) | ** | Astro arc weld #78 | -0.1 | 0.16 | 0.68 | |
| BA271 | 4080 | 365 | 43 | 7.34 | (2.1) | •• | •• | -0.4 | 0.26 | 3.41 | |
| BA280 | 4081 | 37 | 43 | 27.34 | (27.5) | EPR 515 | "O" Ring, | +2.1 | 0.93 | 416.3 ^f | |
| PA281 | 4082 | 65 | 43 | 44.07 | (44.0) | •• | Krytox Coated | +3.5 | 1.62 | 370.0 [£] | |
| | | | | | | | | | | | |

^aProbable error in pretest weighing.

^bCapsule tip had microscopic leak; gas data meaningless.

^CCapsule broke in breaker fixture; gas lost.

^dRate of decomposition is proportional to the area of exposed gold-nickel braze.

^eMylar film dissolved in propellant; area rate is not relevant.

 $^{^{\}mbox{f}}$ Decomposition catalyzed by carbon black used in compounding; area renames are meaningless.

Table 3. Details of Analyses and Results

| Noncondensable Ga | s (N ₂ + H ₂) |
|-------------------|--------------------------------------|
|-------------------|--------------------------------------|

| | Specimen Weight | | Analysis of Propellant ^{a,b} | | | | | Rate | | | | | |
|------------|-----------------|--------------|---------------------------------------|--------|-------------|-----------------------|----------------------|-----------------|---|---|---|-----------|---------------------|
| BAT No. | Test Unit | Initial g | Change g | mg Fe | ppm | н ₂ о % | NH ₃ % | Total cc STP | Uncorrected cc x 10 ⁻³ • day ⁻¹ | Corrected for Control cc × 10 ⁻³ • day ⁻¹ | Specimen Surface Area cm ² | Area Rate | _{cm} -2 |
| BA001 | 4001 | 1.8307 | -0.0004 | <0.02 | < 0.5 | 0.63 | 0.02 | 0.67 | 0.83 | 0.16 | 19.4773 | 0.01 | |
| BA002 | 4002 | 1.8808 | 0.0000 | < 0.02 | <0.5 | 0.66 | 0.02 | 0.45 | 0.56 | -0.11 | 19.4926 | -0.01 | |
| BA003 | 4003 | 1.8674 | -0.0002 | < 0.02 | <0.5 | 0.66 | 0.02 | 1.51 | 1.87 | 1.20 | 19.4926 | 0.06 | |
| BA004 | 4004 | 1.8745 | -0.0001 | < 0.02 | < 0.5 | 0.68 | 0.01 | 0.32 | 0.48 | -0.19 | 19.4773 | -0.01 | |
| BA005 | 4005 | 1.8774 | +0.0025 | <0.02 | < 0.5 | 0.68 | 0.01 | 1.59 | 2.08 | -1.92 | 19.4621 | 0.10 | |
| BA006 | 4006 | 1.8189 | ~0.0002 | < 0.02 | < 0.5 | 0.65 | 0.03 | 4.60 | 6.01 | 2.01 | 19.4926 | 0.10 | |
| BA007 | 4007 | 1.8308 | 0.0000 | ~0.02 | < 0.5 | 0.84 | 0.03 | 2.58 | 3.37 | -0.63 | 19.4926 | -0.03 | |
| BA008 | 4008 | 1.8121 | -0.0003 | < 0.02 | <0.5 | 0.65 | 0.04 | 5.88 | 7.69 | 3.69 | 19.4773 | 0.19 | |
| BA014 | 4009 | 17.9183 | -0.0008 | _c | | 0.58 | 0.02 | 1.15 | 1.43 | 0.76 | 36.55 | 0.02 | |
| BA015 | 4010 | 19.7705 | -0.0012 | - | | 2.95 | 0.01 | 3.52 | 4.36 | 3.69 | 37.59 | 9.16 | |
| BA016 | 4011 | 20.5209 | -0.0009 | - | | 0.56 | 0.04 | 6.57 | 8.14 | 4.14 | 39.57 | 0.10 | |
| BA017 | 4012 | 19.8750 | -0.0005 | 0.125 | 3.1 | 0.86 | 0.60 | 110.57 | 144.54 | 140.54 | 39.92 | 3.52 | |
| BA023 | 4013 | 10.5977 | -0.0004 | 0.02 | 0.5 | 0.63 | 0.10 | 6.17 | 16.32 | 15.65 | 21.1697 | 0.74 | ORIGINAL OF POOR |
| BA024 | 4014 | 9.8477 | -0.0004 | 0.02 | 0.5 | 0.50 | 0.14 | 7.69 | 20.34 | 19.67 | 20.7925 | 0.95 | చ్ టై |
| BA025 | 4015 | 10.4620 | -0.0004 | 0.02 | 0.5 | 0.62 | 0.32 | 32.86 | 86.93 | 82.93 | 21.2021 | 3.91 | POOR |
| BA026 | 4016 | 10.5614 | -0.0001 | 0.02 | 0.5 | 0.70 | 0.27 | 28.31 | 74.89 | 70.89 | 22.5703 | 3.14 | |
| BA034 | 4017 | 0.6948 | -0.0004 | < 0.02 | < 0.5 | 0.52 | 0.02 | 1.95 | 2.34 | 1.67 | 2.7429 | 0.61 | QI |
| BA035 | 4018 | 0.6995 | -0.0003 | < 0.02 | < 0.5 | 8.21 | 0.02 | 5.99 | 7.17 | 6.50 | 2.7040 | 2.40 | 58 |
| BA036 | 4019 | 0.6989 | +0.0155 | < 0.02 | < 0.5 | 0.76 | 0.16 | 35.60 | 42.63 | 38.63 | 2.7392 | 14.10 | |
| BA037 | 4020 | 0.7015 | +0.0071 | < 0.02 | < 0.5 | 0.71 | 0.02 | 9.27 | 11.10 | 7.10 | 2.7068 | 2.62 | PAGE 18 |
| BA045 | 4021 | 2.0661 | +0.0001 | < 0.02 | < 0.5 | 0.57 | 0.02 | 2.18 | 2.61 | 1.94 | 6.61 | 0.29 | |
| BA046 | 4022 | 2.1141 | -0.0003 | < 0.02 | <0.5 | 2.33 | 0.03 | 2.50 | 2.99 | 2.32 | 7.04 | 0.33 | |
| BA047 | 4023 | 2.1348 | -0.0002 | < 0.02 | ≮0.5 | 0.93 | 0.04 | 5.01 | 6.00 | 2.00 | 7.03 | 0.28 | |
| BA048 | 4024 | 2.0952 | 0.0000 | < 0.02 | < 0.5 | 0.90 | 0.08 | 19.80 | 23.71 | 19.71 | 6.80 | 2.90 | |
| BA056 | 4025 | 11.9468 | -0.0010 | < 0.02 | <0.5 | 1.03 | 0.06 | 7.86 | 10.19 | 9.52 | 21.75 | 0.44 | |
| BA057 | 4026 | 12.4409 | -0.0010 | < 0.02 | < 0.5 | 1.21 | 0.18 | 20.57 | 26.68 | 26.01 | 22.06 | 1.18 | |
| BA058 | 4027 | 12.3262 | -0.0011 | 0.050 | 1.3 | 0.70 | 0.89 | 155.32 | 201.45 | 197.45 | 21.93 | 9.00 | |
| BA059 | 4028 | 12.3092 | -0.0012 | 0.125 | 3.1 | 0.86 | 0.74 | 108.45 | 140.66 | 136.66 | 21.96 | 6.22 | |
| BA067 | 4029 | 10.5426 | -0.0007 | _ | | 0.81 | 0.01 | 2.76 | 3.58 | 2.91 | 21.42 | 0.14 | |

Table 3. Details of Analyses and Results (continuation 1)

| | | | | | | | | | | Noncondensable Gas | (N ₂ + H ₂) | |
|--------------|--------------|-------------------------|-----------------|--------|-----------|-----------------------|----------------------|--------|---|---|---------------------------------------|--|
| | | | | Anal | ysis of E | Propellar | nt ^{a,b} | | Rate | | | |
| BAT No. | Test Unit | Specime Initial g | Change | mg Fe | ppm | H ₂ O % | NH ₃ | Total | Uncorrected cc × 10 ⁻³ • day ⁻¹ | Corrected for Control cc × 10 ⁻³ • day ⁻¹ | opecimen Surface Area cm ² | Area Rate cc x 10 ⁻³ · day ⁻¹ · crs ⁻² |
| | | | | _ | | | | | | | ** ** | |
| BA068 | 4030 | 11.2825 | -0.000 8 | _ | | 0.98 | 0.07 | 8.50 | 11.02 | 10.35 | 21.05 | 0.49 |
| BA069 | 4031 | 11.6180 | +0.1422d | - | | 0.86 | 0.20 | 24.00 | 31.70 | 27.70 | 21.59 | 1.28 |
| BA070 | 4032 | 11.5931 | -0.0008 | < 0.02 | < 0.5 | 0.92 | 0.52 | 76.87 | 107.51 | 103.51 | 21.56 | 4.80 |
| BA078 | 4033 | 5.5458 | -0.0004 | < 0.02 | < 0.5 | 1.18 | 0.02 | 1.66 | 2.08 | 1.41 | 26.8885 | 0.05 |
| BA079 | 4034 | 5.6452 | 0.0000 | < 0.02 | < 0.5 | 1.26 | 0.02 | 0.99 | 1.24 | 0.57 | 28.3715 | 0.02 |
| BA080 | 4035 | 5.6520 | 0.0000 | < 0.02 | < 0.5 | 0.52 | 0.11 | 15.23 | 19.40 | 15.40 | 27.0450 | 0.57 |
| BA081 | 4036 | 5.5455 | 0.0000 | <0.07 | < 0.5 | 0.55 | 0.18 | 26.42 | 33.66 | 29.66 | 27.7521 | 1.07 |
| BA300 | 4037 | 5.2560 | -0.0009 | <0.02 | < 0.5 | 0.80 | 0.02 | 1.06 | 1.31 | 0.64 | 20.0694 | 0.03 |
| BA301 | 1038 | 4.9612 | +0.0005 | < 0.02 | < 0.5 | 0.65 | 0.04 | - | - | | 20.0753 | |
| BA302 | 4039 | 4.3396 | -0.0006 | - | | 0.84 | 0.01 | 0.61 | 0.76 | 0.09 | 20.0754 | <0.01 |
| BA303 | 4040 | 4.8056 | -0.0012 | - | | 1.56 | 0.02 | 0.55 | 0.68 | 0.01 | 20.1092 | < 0.01 |
| BA304 | 4041 | 5.1021 | -0.0007 | < 0.02 | < 0.5 | 0.83 | 0.04 | _f | - | - | 20.2661 | |
| BA305 | 4042 | 5.2821 | +0.0006 | 0.02 | 0.5 | 0.65 | 0.02 | 9.37 | 12.25 | 8.25 | 20.3587 | 0.41 |
| BA306 | 4043 | 5.0690 | 0.0000 | < 0.02 | < 0.5 | 0.62 | 0.05 | 7.91 | 10.34 | 6.34 | 20.2475 | 6.31 |
| BA307 | 4044 | 5.0142 | -0.0005 | < 0.02 | < 0.5 | 0.70 | 0.03 | 4.36 | 5.70 | 1.70 | 20.0859 | 0.98 |
| 3A100 | 4045 | 8.3294 | -0.0007 | - | | - | < 0.05 | 0.63 | 3.42 | 2.75 | 21.2098 | 0.13 |
| BA101 | 4046 | 8.3596 | -0.0003 | - | | _ | 0.14 | 3.67 | 14.98 | 14.31 | 21.1961 | 0.68 |
| BA102 | 4047 | 8.3758 | -0.0009 | - | | | 0.10 | 1.10 | 2.90 | 2.90 | 21.1961 | 0.14 |
| BA103 | 4048 | 8.3975 | +0.0002 | _ | | - | 0.04 | 1.33 | 3.64 | 2.97 | 21.1825 | 0.14 |
| BA109 | 4049 | 15.0951 | -0.0007 | _ | | _ | 0.10 | 1.97 | 8.04 | 7.37 | 23.9895 | 0.31 |
| BA110 | 4050 | 14.6483 | -0.0003 | _ | | _ | 0.06 | 3.95 | 10.82 | 10.15 | 24.7093 | 0.41 |
| BA112 | 4051 | 4.6890 | -0.0009 | _ | | - | < 0.05 | 9.32 | 38.04 | 37.37 | 10.1851 | 3.67 |
| EA113 | 4052 | 4.7486 | -0.0004 | _ | | _ | 0.10 | 9.85 | 26.99 | 26.32 | 10.1216 | 2.60 |
| BA115 | 4053 | 2.7318 | -0.0007 | | | _ | < 0.05 | 3.15 | 12.86 | 12.19 | 5.8837 | 2.07 |
| BA116 | 4054 | 2.7818 | +0.0001 | _ | | _ | 0.08 | 0.55 | 1.51 | 0.84 | 5.9570 | 0.14 |
| BA118 | 4055 | 6.0872 | -0.0011 | _ | | _ | 0.30 | 21.89 | 99.50 | 98.83 | 28.58 | 3.46 ⁸ |
| BA119 | 4056 | 5.9381 | -0.0003 | _ | | _ | 0.29 | 38.47 | 136.90 | 136.23 | 27.00 | 5.05 ^g |
| BA120 | 4057 | 5.8159 | -0.0008 | _ | | _ | 0.18 | 34.54 | 100.41 | 99.74 | 27.45 | 3.63g |
| BA121 | 4058 | 5.8271 | -0.0002 | 0.020 | 0.5 | _ | 0.08 | 110.08 | 274.51 | 273.84 | 27.45 | 9. 98^g |
| DAIZI | 4070 | 3.04/1 | -0.0002 | 0.020 | U. J | _ | v. v o | 110.00 | 617.31 | 3/J.GT | 21.73 | /.7 0 - |

ORIGINAL PAGE IS OF POOR QUALITY

Table 3. Details of Analyses and Results (continuation 2)

Noncondensable Gas (N₂ + H₂)

| | | | • | Analy | ysis of I | Propellar | nt ^{e,b} | Rate | | | - | | |
|----------------|--------------|-------------------------|---------|-------|-----------|-----------------------|-------------------|---------------------|---|---|---------------------------------------|--|------------------|
| BAT No. | Test Unit | Specime Initial g | Change | Fe mg | ppm | H ₂ O % | NH ₃ | Total cc STP | Uncorrected cc x 10 ⁻³ • day ⁻¹ | Corrected for Control cc × 10 ⁻³ • day ⁻¹ | Specimen Surface Area cm ² | Area Rate cc × 10 ⁻³ • day ⁻¹ • cm ⁻² | |
| | | • | | _ | | | | | • | · | | 9.15 ^h | |
| BA127 | 4059 | 0.0090 | _ | 0.016 | 0.4 | - | 0.14 | 6.19 | 16.96 | 16.29 | 1.7806 | 9.13 9.43 ^h | |
| BA128 | 4060 | 0.0089 | - | - | | - | 0.07 | 6.31 | 17.29 | 16.62 | 1.7620 | | |
| BA130 | 4061 | 21.7411 | -0.0011 | - | | _ | 0.11 | 2.43 | 7.89 | 7.22 | 13.7489 | 0.53 | |
| BA131 | 4062 | 21.4048 | -0.0013 | - | | _ | 0.08 | 0.48 | 1.41 | 0.74 | 13.5632 | 0.05 | |
| BA133 | 4063 | 0.6402 | -0.0287 | | | - | 0.06 | 15.58 | 50.58 | 49.91 | 1.38 | 36.17 | |
| BA134 | 4064 | 0.6172 | +0.0008 | - | | - | 0.09 | 5.51 | 15.10 | 14.43 | 1.38 | 10.46 | |
| SE200 | 4065 | 12.1607 | -0.0005 | - | | _ | <0.05 | 6.41 | 20.81 | 20.14 | 22.4651 | 0.90 | |
| SE201 | 4066 | 11.7659 | +0.0001 | - | | _ | 0.04 | 0.90 | 2.41 | 1.80 | 22.2418 | 0.08 | |
| SE210 | 4067 | 10.6931 | 0.0000 | _ | | _ | 0.05 | 0.55 | 1.51 | 0.84 | 22.07 | 0.04 | 00 |
| SE211 | 4068 | 10.9716 | -0.0011 | _ | | _ | < 0.05 | 0.24 | 1.30 | 0.63 | 22.15 | 0.05 | 유용 |
| SE220 | 4069 | 6.8595 | -0.0024 | _ | | _ | < 0.05 | 1.05 | 5.71 | 5.04 | 19.5243 | 0.26 | ORIGINAL OF POOR |
| SE221 | 4070 | 6.8879 | +0.0006 | _ | | _ | 0.04 | 1.36 | 3.73 | 3.06 | 19.5243 | 0.16 | ö≨ |
| SE230 | 4071 | 12.1497 | -0.0004 | _ | | _ | < 0.05 | 0.24 | 0.78 | 0.11 | 22.07 | | |
| SE231 | 4072 | 11.9301 | -C:0004 | _ | | _ | 0.64 | 0.35 | 0.96 | 0.29 | 22.22 | 0.01 | 22 |
| SC240 | 4073 | 6.7289 | -0.0003 | _ | | _ | < 0.05 | 3.13 | 10.16 | 9.49 | 88.23? | 0.11 | PAGE :3 |
| SC241 | 4074 | 6,7360 | -0.0008 | _ | | _ | 0.07 | 3.46 | 9.48 | 8.81 | 88.232 | 0.10 | 5 6 |
| BA250 | 4075 | 12./079 | -0.0012 | _ | | _ | 0.06 | 1.12 | 4.57 | 3.90 | 22.2552 | 0.18 | 78 |
| BA251 | 4076 | 12.7510 | -0.0002 | _ | | _ | 0.05 | 3.10 | 8.49 | 7.82 | 32.1961 | 0.35 | |
| BA251 BA260 | 4077 | 7.1138 | -0.0002 | _ | | | < 0.95 | 1.00 | 4.08 | 3.41 | 21.21 | 0.16 | |
| | | | | | | | | | 1.43 | 0.75 | 20.95 | 0.04 | |
| BA261 | 4078 | 7.0385 | -0.0002 | - | | - | 0.04 | 9.52 | | | | 0.68 | |
| БА270 | 4079 | 6.7365 | -0.0001 | - | | - | 0.15 | 3.24 | 13.22 | 12.55 | 18.56 | | |
| BA271 | 4080 | 6.6650 | -0.0304 | 0.C23 | 0.6 | - | 0.18 | 23.40 | 64.11 | 63.44 | 18.58 | 3.41 | |
| PA280 | 4081 | 0.5132 | +0.0021 | 6.010 | 0.3 | 0.56 | 0.61 | 98.60 | 2664.9 | 2664.2 | 6.40 | 416.3 ⁱ | |
| BA281 | 4082 | 0.5141 | +0.0035 | 0.010 | 0.3 | 0.55 | 0.40 | 153. 9 6 | 2368.6 | 2367.9 | 6.40 | 379.0 ⁱ | |

a Based on actual weight of propellant in capsule.

b Halide level undetectable, i.e., < 0.02 mg.

c - Not measured; data not available.

d Probable error in pretest weighing.

e Capsule tip had microscopic leak; gas data meaningless.

f Capsule broke in breaker fixture; gas lost.

g Rate of decomposition is proportional to the area of exposed gold-nickel braze.

h Mylar film dissolved in propellant; area rate is not relevant.

i Decomposition was catalyzed by carbon black used in compounding; area rate values are meaningless.

Table 4. Summary of Analysis of Hydrazine Controls

| | | | | | | | Pr | opellent ^d | | |
|------|---------|----------------------------------|---|-----------------------------|-------------------|-------------------|----------------|---|---|------------------|
| | | | | | Anal | ysia b | | Ner | -Condensable Gar (N ₂ + | H ₂) |
| Test | Days on | Test Temp., ⁰ C | Capuale Pressure at Test Temp., N/cm ² | (Strain Gage Reading) | H ₂ O. | NH ₃ . | Decamposition. | Total cc STP | Pate cc x 10 ⁻³ · day ⁻¹ | Average |
| Unit | Test | | | | _¢ | < 2.05 | < 0.05 | 1.93 | 4.68 | |
| 4100 | 220 | 43 | 0.81 | (6.0) | | 0.04 | 0.04 | 0.26 | 0.71 | |
| 4101 | 365 | 43 | 0.75 | (0) | 0.74 | 0.02 | | _d | - | |
| 4102 | 549 | 43 | - | (0) | 0.72 | | - | €¢.25¢ | - | |
| 4106 | 729 | 43 | - | (0) | 0.70 | 0.01 | 0.01 | 0.45 | 0.62 | 0.67 |
| | 729 | 43 | 0.67 | (0) | 0.71 | 0.01 | 0.01 | • | | |
| 4108 | 127 | | | | | < 0.05 | < 0.01 | 2.98 | 9 45 ¹ | |
| 4103 | 220 | 60 | <1.0 | (U) | - | | 0.11 | 1.26 | 3.14 | |
| 4104 | 401 | 60 | 2.21 | (0) | 0.56 | 0.10 | 0.03 | 2.45 | 4.48 | |
| | 547 | 60 | 2.00 | (0) | 0.72 | 0.02 | | 2.82 | 3.87 | |
| 4105 | | 60 | 1.71 | (0) | 1.55 | 0.01 | 9.02 | | 4.46 | 4.00 |
| 4107 | 729 | | 2.17 | (0) | 0.65 | 6.01 | 9.02 | 3.25 | | |
| 4109 | 729 | 60 | 2.27 | | | | | | | |

Bescd on 40.0 or hydrazine.

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b Metals and halides undetectable.

C- Not measured; data not available.

dCapsule broke in test fixture; gas lort.

classifficient quantity of gas to measure.

¹Value not included in average.

SECTION VI

REFERENCES

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- 6. Adams, R. O., "Review of the Stainless Steel Surface," <u>Journal of Vacuum</u> Science Technology, Vol. 1, No. 12, 1983.

APPENDIX A

PRETEST ANALYSIS OF HYDRAZINE

The pretest analysis of Drum 98367 indicated a very high purity which even meets the requirements of the current MIL-P 26536C, Amendment 2, High Purity Grade. The drum was from JPL's supply of hydrazine at ETS and was chosen because of the low CO_2 content. The full pretest analysis is presented in the report form contained in this appendix.

The handling of the propellant during the filling operation, and the process of removing it after completion of the storage period, can influence the CO₂ content through inadvertent exposure to air. A special test capsule was designated as a CO₂ control capsule. It was filled as part of a regular series of capsules being processed. After freezing and thawing, this control capsule was opened and the propellant removed by the standard procedure. The CO₂ content was determined to be 15 ppm, slightly higher than the 9 ppm in the original hydrazine, but considerably lower than the 30 ppm iimit in the above-mentioned military specification. Although this determination was not normally part of the posttest procedure, two other test units were analyzed for CO₂: 4081 contained 5 ppm CO₂; 4082 contained 19 ppm CO₂.

The data from these three capsules indicated that the procedures employed in filling the capsule were adequate to maintain the desired low CO₂ content. The greatest risk for exposure to air occurs during the opening of the capsule and removal and transfer of the hydrazine to the analysis vial.



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| (PREPARED BY) | (DATE) | (REPORT NO.) | |
|---------------|-------------|-----------------------|--|
| Toth | 4 Dec. 197 | 79 79×10201 | |
| Assay By | (DATE) | PROJECTI | |
| Taylor/Moran | 10 Dec. 197 | 79 NaH, Compatibility | |

TITLE

| Constituent or Property | Results | Specification |
|--|---------------------|---------------------|
| Hydrazine assay, % by weight Note 1 | 99.4 | 98.52 min. |
| Density at 298 K (77°F), g/cm ³ | 1.004 | |
| Particulate, mg/cm ³ | 0.0007 | 1 milligram/l liter |
| Water plus soluble impurities, | 0.62% | 1.0% max. |
| % by weight | | |
| Major impurities, % by weight | | |
| Ammonia (NH ₃) | < 0.12 | C.4% max. |
| Aniline (C ₆ H ₅ WH ₂) | None detected, n.d. | 0.5% max. |
| Toluene (C6H5CH3) | n.d. | |
| Carbon Dioxide (CO ₂) | 0.0009 | 50.0 ppm max. |
| UDMH | n.d. <0.1% | 1 |
| Other | | 1 |
| Sulfated Ash, 2 by weight | < 0.0005 | |
| Atomic Absorption Analysis | | |
| of ash | | |
| Dissolved metals, Aug/g N2H4 (ppm) | | |
| Iron | 0.12 | |
| Aluminum | < 0.1 | |
| Nickel | 0.15 | |
| Manganese | < 0.03 | |
| Cobalt | < 0.03 | |
| Chronium | < 0.01 | |
| Copper | < 0.03 | |
| Zinc | 0.03 | |
| Silicon | < 0.1 | |
| Magnesium | 0.05 | |
| Sodium | 0.05 | |
| Calcium | 0.3 | |
| Barium | < 0.1 | |
| Boron | < 10 | |
| | 0.03 | 1 |

| CRISMAL | 17.35 | S |
|---------|-------|---|
| | | |

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|--------------|-----------------------------|-----------------|
| | Inc. 14 (REPORT NO.) | |
| | | |
| , By | (DATE) PROJECTI | |
| .avlor/Morso | 10 Dec 1979 N.H.Compatibili | tv |

TITLE

ASSAY-HYDRAZINE JPL Drum H8367

| Constituent or Property | Results | Specification |
|---|---|---------------|
| Dissolved amions, µg/g Fluoride Chloride Sulfate Nitrate Nonvolatile residue, mg/cm ³ | n.d. < 5 ppm n.d. < 1 ppm n.d. < 5 ppm n.d. < 5 ppm < 0.005 | 5.0 ppm max. |
| Identification/History Specification Storage Container | Hotes 2, 3 JPL Drum H8367 | Note 1 |
| Test Sample (250 ml size) | 2 bottles | |

NOTES OR REFERENCES

- Hydrazine must conform with Bell Aerospace specification 8803-947047
 Revision A. Chemical composition requirements listed in Section 3.2
 CO₂ requirement of 50 ppm maximum is critical.
- 2. W/A 4078-1 and shipper E6076; hydrazine received 7 December 1979.
- Purified or refined grade hydrazine used on MASA-JPL flight projects
 "Voyager 1977", and "Mars Viking Lander 1975". Hydrazine manufactured by
 Martin Marietta Corp., Denver, Colorado, their specification STM NO20,
 during CY 1973-1975 period.



APPENDIX B

DETAILED LOGS OF ALL TEST UNITS

Log I is a listing of specimens grouped by specimen (BAT) numbers and test unit (JPL) numbers in ascending order. The "Material Description" column also lists the CPR number and the material scheduled for storage. The "Test Duration" column under "Cell" gives the dates of storage at test temperature. The "Refrigeration" column lists the dates for posttest storage in the freezer before analysis. The "Analysis Document" column lists the JPL internal memoranda reporting results of analysis. The "Remark" column lists test temperature (43°C unless otherwise noted), and other information.

Log II is a listing of specimens by ascending capsule number. The "Capsule" column also includes the total internal volume of each capsule. The "Material Description" column lists the date and time of capsule filling. The capsules were then kept in a freezer until the date shown in the "Cell" column, i.e., the beginning of the storage at test temperature. The "Remarks" column lists the volume of hydrazine placed in each capsule.

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| Report Number 79X07500 |
|------------------------------------|
| Project Hydrazine Compatibility |
| Classification <u>UnClassified</u> |
| |

JET PROPULSION LABORATORY CALIFORNIA INSTITUTE OF TECHNOLOGY

SUMMARY

HYDRAZINE MATERIAL COMPATIBILITY TEST SPECIMEN/CAPSULES

| Prepared by | L. R. Toth | Date | July 1979 |
|-------------|------------|------|-----------|
| | | Date | |

APPENDIX OR REVISION

| Dete | Peges Affected | Appendix OR Revision | Remorks | Changed by |
|-------------|----------------|----------------------------|---|---------------|
| 28 May 1980 | 14 | (A) | Terminate test units: 4081 (SE 280); 4082 (SE 281) | L. Toth |
| | |] | | İ |

| NUM | Ber | TES MARROTAL MA | WINS | TEST D | URATION | ANALYSIS | | | Ę | 3 |
|----------|---------|--------------------------|---------|-----------------|-------------------------|----------------------------|---------|-----------|---------|----------|
| SPECIMEN | CAPSULE | MATERIAL DESCRIPTION | | CELL | REFRIG- ERATION | DOCUMENT, IOM, etc. | REMARKS | " | EVISIC | 700 |
| BAT No. | | CAR No. | IN | /OUT | IN/OUT | DATE | | ╛╹ | 25 | 153 |
| 400/ | 7903 | 304L CRES DIAPHRAGM | 24 28 3 | | 14 APR 82 13 MAY 82 | | | | | |
| BA 001 | | / | · / / - | 10.99 | <i>5</i> 0 | 9 Sept 82 | |] } | | |
| 4002 | 7924 | 304L CRES DAPHRAGM | 24 283 | an 80 | 11 MAY 82 | 34411 87 704 9 Sept 82 | | 1 | | |
| BA 002 | | | | ا ج | 28 | | |] | | <u> </u> |
| 4003 | 7929 | 304L CRES DIAPHRAGM | 21 283 | "An 80 | 14 APR 82 12 MAY 82 | 3444T-82-204 9 Sept. 82 | | Bydrazine | DATE) | ATAL |
| BA 003 | | , | - 1 | ا ر | 29 | | | ine | | 1979 |
| 4004 | 7947 | 304 L CRES DIAPHRAGM | 1 | Jan 80 | 14 APR 82. 14 MAY 82 | 344AT-84-204 | | Monopro | 3 | |
| 8A004 | | , | | 5 1 | 31 | 9 Sep + 82 | | bid | Ę | 79X07500 |
| 4005 | 7921 | 3041 CRES : DIAPHRAGM | | an 80 nan 82 | 3 MAR 82 30 MAR 82 | 344AT-82-117 13 May 82 | 60°C | | JPL 1 | 8 § |
| BA005 | | , | 1 9 | 165 | 28 | • | | ହ | Propos | Ì |
| 4006 | 7902 | 304 L CRES DIAPNRAGM | 24 78 J | an PO | 3 MAR 82 31 MAR 82 | 344AT-82-117 13 May 82 | 60°C | 7 | | |
| 81006 | | / | 7 | 165 | 29 | · · | | | 90-98 | |
| 4007 | 7932 | 3041 CRES S | | an 80 | 3 MAR 82 18 MAR 82 | 344A182-117 | 60°C | DRUM H | 965 rev | |
| BA 007 |] | , | . | 765 | 16 |] ' ' | | H | | |
| 4008 | 7936 | 3041 CRES DIAPHRAGM | 24 283 | an 80 | 3 MAR 82 17 MAR 82 | 344A7 82-117 | 60°C | 0367 | | |
| BA008 | | / | | 2.5 | 15 | | |] | | |

| | | 763 | 1 | | | | | + | | _ | _ |
|---------------|----------------|-----------------------|------|------------------------|-------------------------------|------------------------------------|-----------------------------------|-----------|----------------------|----------------------------|---------|
| NUMB CIMEN | BER CAPSULE | | NTHS | TEST D | URATION REFRIG- ERATION | ANALYSIS DOCUMENT, IOM, etc. | REMARKS | | REVISIO | 707 | PREPARE |
| 7 No. | | CPR No. | | IN/OUT | IN/OUT | DATE | | | 1 2 | 7 | į |
| 09 | 7937 | 304 L CRES 2 LINER | | 18 Jan 80 | 14 APR 82 4 MAY 82 | 344AT-82-204 8 Sept.82 | | | | | |
| 014 | | 2 | | EC | 21 | ' | | | 1 | | 1 |
| 010 | 7955 | 304L CRES & | 24 | 28 Jan 80 | 14 APR 82 4 M.94 82 | 344AT-82-204 350pt 91 | | | | 1 | |
| 015 | | 2 | | . Jan 1 | 21 | | | | | L | |
| 7// | 7912 | 30 FL CRES 2 | 1 | 285an 80 | 14 APR 82 10 MAY 82 | 344AT-83 204 8 Sept 82 | 60°C 42.0 CC N2H8 | Hydr | DATE | ولول | DATE |
| 016 | | 2 | ľ | W- 20.00 | 27 | | | Hydrazine | | ¥ 197 | |
| 2/2 | 7938 | 304 L CRES 2 LINER | 2# | 28 Jan 80 03 Man 82 | 3 MAR 87 26 MAR 82 | 344AT-87-117 13 May 82 | 60°C | H g | 3 | 9 79 | 2 |
| 017 | ! | 2 | | 765 | 24 | , | | oprop | DIECTI | 79 X 075 0 0 | P TROS |
| 0/3 | 79202 | 347 CRES 2 DISC | 24 | 18 MAR 80 | 1 464 21 | 244.AT-81-061 22APA 81 | Special companie 1.25" di | | 171 Mete | 8 | 5 |
| 023 | | 3 | | 378 OH5 | 6 PAYS | | | r P | rial Com Proposal | | |
| | 79204 | 347 CRES 2 DISC | | 18 Mar 80 1 200 :1 | 2767.21 1767.21 | 244-4J-81-061 22/8-81 | special capsule 1.25" die | | | | |
| 032 | | 3 | | 25K 2000 | GDAYS | | | l | 90-965 90-965 | l | |
| 015 | 79205 | 347 CRES DISC | | 18 Mar 80 | 1.4802 XI | 344-di-81-04 22100 81 | Special rapsula 1.25" dia 60°C | | 0-965 rev | | |
| 025 | | 3 | - 1 | | くいかい | | | | N | | |
| | 79206 | 347 CRFS DISC | | 18 Har 80 AOD S. | 1 AC 11 | 344 / 1. 11 CM | Special capsule 1,25" dia 60°C | | | | |
| 016 | | 3 | | | SIMI | | | | | | |

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| | 릙 | | ANALYSIS | URATION | TEST D | TEST | | BER | NUM |
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| TOT TOTAL | | REMARKS | DOCUMENT, IOM, etc. | REPRIG- BRATION | CELL | | MATERIAL DESCRIPTIO | CAPSULE | SP E CIMEN |
| 753 | ļ | | DATE | IN/OUT | IN/OUT | | CPR No. | | BAT No. |
| | | | 344AT.82.205 8 Sept 82 | ;2 MAY 82 25 MAY 82 | 385an80 12 MAY&X | 21 | PLUG | 7926 | 4017 |
| | Ì | | ! | 14 | 236 | | 4 | | BA 034 |
| | 1 | | 344AT-82-205 8 Sept B2 | 12 MAY 82 25 MAY 82 | 28 Jan 80 | 24 | 304L CRES PLUG | 7933 | 4018 |
| <u> </u> | _ | | · | 14 | 826 days | | 4 | | BA 035 |
| (BATE) | | 60 °C | 9 Sept 82 | 30 JUNE 82 | | 24 | 304L CRES PLUG | 7925 | 4019 |
| 1979 | | | | 50 | 8.36 | | 4 | | BA 036 |
| ᆲ | | 60°C | 344AT 87-205 8 Sept 82 | | 28 Jan 80 12 MAY 42 | 24 | 3041 CRES PLUG | 7935 | 4010 |
| 79X07500 | | | | 50 | 824 | | 4 | | BA 037 |
| | | | 344AT-81-205 8 Sept 82 | 27 444 (4) | 28504 80 12 M482 | 24 | 304 L CRES WELD TIG | 7940 | 4021 |
| E | | | | 12 | 836 | | 5 | <u> </u> | BA 095 |
| 6 | | | 344A7-82-205 8 Sept 82 | | 1 | 24 | 3041 CRES WELD TIG | 7944 | 4012 |
| | | | | 16 | 436 | | 5 | | BA 046 |
| TIG9 | | 60°C | 344AT 82-205 8 Sept 82 | | 285an 80 | 24 | 3041 CRES WELD TIG | 7911 | 4023 |
| 1 | - {- | | | 49 | ્રુકિ. | | 5 | | BA 047 |
| | | 60°C | 95 pt 9- | 17 MHY 82 1 JULY 82 | | 29 | 304L CRES WELD TIG | 7928 | 4024 |
| | | | | 51 | | | 5 | | BA 048 |

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| NUM SPECIMEN | BER CAPSULE | MATERIAL ^ DESCRIPTION | FS T ION TH | TEST D | PURATION REFRIG- ERATION | ANALYSIS DOCUMENT, IOM, etc. | remarks | Thur. | REVISION | 7 | |
|-----------------|----------------|---------------------------|----------------|--------------|---------------------------|------------------------------------|---------------------|-----------|--------------------------|----------|-------------|
| BAT Na | | BAT CPR No. | | IN/OUT | IN/OUT | DATE | | _ | 2 | N | |
| 4025 | 7971 | 30 \$ L CRES | 24 | 4 Mar 80 | 14 APP 82 | 344AT-82.204 | | | İ | 73 | |
| | | WELD EB # 40 7 | | 12.71. | 11 MAY 82 | 8 Sopt 92 | | 1 | | | 1 |
| BA 056 | | 407 | | 2011 | 28 | | | 1 | | | 1 |
| 4026 | 7960 | 3042 CRES | 24 | 4 Mar 80 | 14 APR 82 | 344AT-82-204 | | 1 | | | ı |
| / 000 | , , , | WELD EB | | y ans | 12 MAY 82 | 8 Sept 82 | | | | | ı |
| BA 057 | | # 407 | / | 1 27 | 29 | İ ' | | 1 | | | |
| | 7922 | 304LCRES | 21 | | 3 MAR 82 | 344AT-82-117 | 60°C | - | Ş | . 9 | 1 |
| 4027 | 1742 | WELD EB | ~ ′ | | 1 ' | 8 Sept 92 | 100 6 | Bydrazine | | (BATE) | |
| | | #407 | | | 24 | 1 ' | | | 1 | | ı |
| BA 058 | | 6 | | 1729 | | 71111 47 47-17 | | - F | ł | 1979 | ı |
| 4028 | 7950 | 304L CRES WELDEB | 24 | 18 HAL 80 | 30 MAR 82 | 344AT-82-17 | 60 °C | ğ | 3 | 7 3 | |
| | | #407 | | | 27 | 13 May 82 | | Monopro | Į | 8 2 | |
| 84 059 | | 6 | | 115 | | | | | 3 | 79307500 | |
| 4029 | 7946 | 304L CRES | 24 | 4 Mar 80 | | 344A7-92-204 | | pellant | | ع حا | 4 |
| | , , | WELD EB | | 14-20008 | 14 MAY 82 | 4 Sept 82 | | | | | H |
| BA 067 | | 7 | | 171 | 31 | | | ន | Propos | | ı |
| 4030 | 7965 | 30 FL CRES | 21 | 1 Mar 80 | 14 APR 82 | 344AT-82-204 | | Grade* | 0 C | 1 | |
| 7030 | 1103 | MELD EB | | 11 115 | 12 MAY82 | 8 Sept 82 | | * | | | |
| BA 068 | | # f06 | | 1.9 | . 29 | | | | etibility 90-965 rev. | | |
| | 7958 | 304L CRES | 14 | 18 Man 80 | 14 APR 82 | 344A1 82-204 | 60°C | 1 | SE |] | ١, |
| 4031 | 7700 | WELD EB | | 401 3 3 3 | 10 MAY82 | | | 1 | 2 2 | 1 | į |
| | | #406 | | 200 | 27 | ' | | 1 | 7. 2 | | |
| 84 069 | | 30fl CRES | 24 | 19 Mas. Dr | 2 mag 22 | 344AT-82-117 | 60°C | 1 | ~ | | |
| 4032 | 7939 | WELD ER | | cis, kan'i k | 25 NAR 82 | | 80.5 | 1 | | | 9 |
| | | #406 | | 41.2 | 23 | 13 May 42 | | 1 | l |] | \parallel |
| BA 070 | | 7 | | L | l <u></u> | | <u></u> | Į | 1 | 1 | |
| | | *Specifi | catio | n: Bell Aer | ospace Textr | on; Report No. | 8803-947047, rev. A | 1 | ļ | 1 | |

| | UMBER | 7 | F37 m | | | | |
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| SPECIME BAT No | 1 | BAT MATERIAL MA | CEL | -01410- | | | 3 8 3 |
| 4033 | | GPR No. | IN/OUT | ERATION IN/OUT | IOM, etc | · | EVISION OF THE PARTY OF THE PAR |
| | 1,,0, | 308L/304L CRES RING - DIAM | | 80 12 MAY 8 | DATE 12 3444T-82 - 20 | 5 | |
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| | 1 | RING-DIAPH. | 12 mays | 26 may 82 | 12 02.2) | 5 | |
| BA 079 | | 8 | | 26 MAY 80 | 8 Sept 22 | 1 | 1111 |
| 4035 | 7966 | 3081/3041 5265 | | 9 12 2011 2 | | 1 | ; |
| 1_ | 1 | RING-DIAPH. | | 80 12 MAY 82 | 12. 1 2. 203 | 60°C | _# |
| BA 080 | | 18 | 206 | 30 | 8 sep + 82 | 1 - 5 | (DATE) |
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| | | RING-DIAPH. | | 0 12 MAY 82 | 3441-82-205 | 60°C | |
| BA 081 | | a | 785 | CT DUNE OZ | 8 Sept 82 | 100 6 | |
| 4037 | 7914 | 30 \$L CRES 11 | | 49 | | _ | (REPORT NO.) 79X07500 PROJECT! H J |
| | | LINER | 1-12-4 60 | 1 APR 82 | 344 A T-82-204 | Replace 84 085 | |
| BA 300 | | q | | 13 MAY 82 | 8 Sept 82 | 1 | Mater JPL P |
| 4038 | 7945 | 309L CRES | .50.00 | 30 | • | 1 | Material JPL Propo |
| 1 | | LINER | 03 May | 3 MAR 82 - | 344A7-82-117 | Raplace 81 086 | Proposal |
| BA 30/ | . | 9 | 0/ | 23 MAR 82 | 13 May 82 | 24076 | |
| 4039 7 | 754 3 | 304L CRES 24 | 28 Jan 80 | 25 | · 1 | | |
| . 1 |]4 | LINER | 1 1 | 1 17 17 17 16 14 | 44AT-82 -20H | Replace BA 087 | 90-965 rev. |
| 84 302 |] | 9 | 112-6 V | שש צירויי כ | 8 Sept 82 | | |
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| R4 303 | 9 | , j | | | 8 Sept 52 | | |
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| | | "specification | : Bell Aero | space Textron | : Penane N. a | 803-947047, rev. A | 1 1 11 |

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| NUM | ER CAPSULE | Test Material Months | CELL D | URATION REFRIG- | ANALYSIS DOCUMENT, IOM, etc. | remarks | J.F. | VISION | 767 |
|----------------|---------------|---------------------------|------------------------|-----------------------------|------------------------------------|-------------------------|------------------|-------------|---------------|
| BCIMEN | CAPSULE | DESCRIPTION BAT CPR No. | IN/OUT | ERATION IN/OUT | DATE | Replace BA 089 | | , | £ 3 |
| 47 No. L041 | 79/8 | 304L CRES 24 21NER | 28 Jan 80 Ozman 82 | 5 MAR 82 50 MAR 82 27 | 13 May 82 | 60°C | | | |
| 4 304 | | 9 | 7105 | 2 200 40 | 2 MAT-82-117 | Replace BA 090 | | | |
| 1042 | 7927 | 304 L CRES 24 LINER | 28 Jan 80 07 mar 82 | 3 MAR 82. 19 MAR 82 | 1 | 60°C | | | |
| | | | 165 | 17 | | 0 1 01 101 | —— ₉ | 9 | (BATE) |
| BA 305 | | 3041 CRES 24 | 21 Jan 80 | 3 MAR 82 | 344AT-82-117 | Replaces BA 091 | di | 1 | |
| 4043 | 7943 | LINER | D3 Man 87 | 19 MAR 82 | · · | 1 | Bydrazine | | (DATE) (DATE) |
| BA 306 | | 9 | 765 | 8 MAR 82 | 244AT-82-117 | Replace 84 012 | * | | |
| 4044 | 7930 | 304L CRES 24 | ozmanss | 3 MAR 82 17 MAR 82 15 | 13 Ma/80 | 60°C | Honopro | ROBCII | 79X07500 |
| M 307 | 1 | 9 | 706 | | | | | 当点 | ၂႘ န |
| 40 45 | 7982 | A186 CRES TANK SHELL | 4 SEPT 80 | 15 OCT 80 | 344 AT- 20-16 | | ellant G | JPL Proposi | |
| | | 10 | 184 gang | 41 OA1 S | , | | Grade* | 1 5 5 E | ;] |
| 40 + 6 | 7920 | A 286 CRES TANK SWELL | 4 MAN 80 | 14 MM BD | 3086 80 | 1 | | 1 90-965 re | |
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| 404 | 3 7959 | A186 CRES TANK SHELL | 4 MAR 8 | 1 hong? | '' I' - A.A.I CI | | | | |
| BA 10 | 3 | 10 | 36574 | 15 <. | Pennet 1 | 4o. 8803-947047, rev. A | | | |

| SPECIMEN | Capsule | MATERIAL MONT | | REFRIG- ERATION | AMALYSIS DOCUMENT, IOM, etc. | REMARKS | | REVISION | 76 |
|----------|---------|-----------------------------------|---------------------------------|-----------------------|------------------------------------|-------------|-----------|------------------------|----------|
| BAT No | | BAT CPP No. | IN/OUT | IN/OUT | DATE |] | | ~ | 13 |
| 4049 | 7963 | A286 CRES WELD TIG #411 | 4 Mar 80 | 4 NOU 80 | 344.4T. 70.173 50EC 80 | | | | <i>H</i> |
| BA 109 | | 11 | 245 Aay | CHADI |] | 1 | 1 | ļ | i |
| 4050 | 7981 | A286 CRES WELD TIG # 4!! | 1 | 4 MAR 81 23 AR 81 | 344AT-81-086 15 MAY 81 | | | | |
| BA 1.0 | | // | 765 DAYS | 1 | <u> </u> | | | | |
| 1051 | 7974 | A 286 CRES & WELD TIG # 417 | प्राच्या १०० | 20 NOU 80 | 7,04-AT- 80-173 5 DEC 80 | | Bydrazina | | July |
| BA 112 | | /2 | 342 Bring | 16 DAYS_ | <u> </u> | | | i i | 1979 |
| 4052 | 7977 | A 286 CRES 6 WELD TIG # 417 | 3 Mar 80 4 Mar 81 365 Day | 49 49 | 344 AT - 81 066 15 And 1 81 | | Monoprop | <u></u> | 79X07500 |
| 4053 | 7934 | 308L CRES 6 | | 14 400 %O | 344-AT-50-173 | | py 11ant | 77 X 60 | 8 5 |
| 8A 115 | | /3 | 245 8-45 | 1 | , , , , , , | | nt Gr | Material JPL Prop | |
| 4054 | 7951 | 3081 CRES & | 9May 80 4 may 81 | 4 mar 81 24 Apr 81 | 344-18-81-81 15144 81 | | ad e | rial Com | |
| A //6 | | /3 | 365day | 1 2 | | | | 90-965 | |
| 7055 | 7915 | 3084/3044 CRES & | | 4 SEPT 80 | 7410 80 | 42 CC No HA | | tibility 0-965 rev. | |
| BA 118 | | 14 | 220 Dags | 43 0415 | | | | . 2 | |
| 4056 | 7923 | 3081/3091 CRES & | 4 1100 S | 1 | 500 KD 70 M | 42 CC N. He | | | |
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| CIMEN CIMEN | er Capsule | Test MATERIAL MONTH, DESCRIPTION BAT | | REFRIG- BRATION | ANALYSIS DOCUMENT, IOM, etc. | REMARKS | T. | TETATE | 75 | GERVERA |
|-------------|---------------|--------------------------------------|-----------------------|--------------------|------------------------------------|---|--------|-------------|------------|---------|
| T No. | | CPR NA. | IN/OUT | IN/OUT | DATE | | | 3 | | 2 |
| 057 | 7941 | 308L/304L CRFS 6 LINGR-DIAPH. | 6 MN 8/ | 23 MAK! | 344 AT- 51-04 | | | | H | 3 |
| 120 | | 14 | 344 DAYS | ב דיפס רו | 1 | | ł | l | ĺ | i |
| 058 | 7953 | 308L/304L CRES 6 LINER-DIAPN. | 2850n 80 4 Mar 51 | 21 Apr 81 | 344AT-81-096 15 MAN 81 | | | | | |
| 121 | | 14 | 401 Days | 48 | | | | | L | |
| 059 | 7964 | MYLAR FILM 6 TYPE 6 DISC | 1 May 80 4 may 81 | 25 Mar 81 | 34481-81-084 15 444 81 | | Bydras | (a) (A) | July | (BTAG) |
| 127 | | 15 | 365 Days | اہر | ł | | | ı | 1979 | |
| 060 | 7968 | MYLAR FILM 6 TYPE G DIJC | 4 May 80 4 May 81 | 30Apr 81 | 15 HAY 81 | | Honor | 980 | 9 79307500 | (MEPO |
| 128 | | 15 | 365 Dans | 57 | | | ğ | [3 | 2 | 3 |
| 061 | 7972 | 304L CRES 6 WELD EB | 6 3 AM 81 | 21 244 81 | 344.AT-81-09 13 FEB 81 | | ellan | 12 15 | 8 | ٤ |
| 130 | | #404 | 30 & D412 | 15 0445 | <u>,</u> | | 9 | | | |
| 062 | 7998 | 304L CRES 6 WELD EB #404 | 29 Mar 80 4 Mar 81 | NAGA. | 15 MAY 61 | a capable 7979 bioben L. capable 7980 broken c. apaimen recleaned a L | rade* | Proposal 90 | | |
| /3/ | | 16 | 34000 | 48 | 4 | | | 8 5 | l | |
| 063 | 7975 | SAMARIUM 6 COBACT MAGNET | 4 May 80 4 JAN 81 | 51 2411 XI | 13 F8381 | | | 90-965 rev. | | |
| 133 | | 17 | 308 DAYS | 150445 | | | | ~ | l | Å |
| | 7976 | SAMARIUM 6 COBALT MAGN-T | 4 Mar 80 4 Mary1 | 29 Apr 8) | 344AT-31.0% 6 15 MAY 81 | | | | | |
| 134 | | 17 | 3658am | ٠. ٧٠ | } | | | | | H |

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| 3 8 | | | ANALYSIS | URATION | TEST D | TEST MANTE | BER | HUNG |
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| • OTSTO | - | REMARKS | DOCUMENT, IOM, etc. | REFRIG- ERATION | CELL | DESCRIPTION | CAPSULE | SPECIMEN |
| 2 | } | | DATE | IM/OUT | IN/OUT | SAT CPR NA. | | BAT No. |
| | | | 344AT-41-019 13 FEB 81 | c samel | 4 Mar 80 6 Jan 81 | 17-4 PH CRES 6 H 1050 ELECTRO POLISH | 7948 | 4065 |
| - 1 | | | | 17 0445 | 366 0002 | 12 | | SE 200 |
| | | | 344AT-81.086 | 1 May of | 4 Mar 80 4 mar 31 | 17-4 PH CRES (HIOSO ELECTROPOLISH | 7973 | 4066 |
| | } | | | 580 | 365 days | 18 | | SE 201 |
| DATE) | Bydrazine | | 34487-81-086 15 MA 81 | 26Man 81 | | 17-1 PH CRES HIOSO ELECTRO POLISH CHROME PLATE | 7961 | 4067 |
| 1 1 | 18 | | | | 3658aug | /2 | | S€ 2/0 |
| (TOBLOPED | Monop | | 344-27-70-160 7/170 80 | 16 oct 80 | 4 5807 80 | HIOSO ELECTRO POLISH | 7967 | 4068 |
| 3 | jā | | | 42045 | 184 Dank | CHROME PLATE | _ | SE 2/1 |
| | 1 | Spacial regards mach 6.775 dia | 7 MOU 80 | 4 SEPT 80 | 4 5487 80 | 17-9PH CRES 6 CN 900 SPRING | 7986 | 4069 |
| Proport Trial | 5 | | | 43045 | 1842 | 20 | | 3e 320 |
| | 2 | special companie mark 0.8% dia | 344AT - 8/086 15MAY 81 | 20APA 81 | 4 Mar 80 4 Mar 41 | 17-9PN CRES 6 CH 910 SPRING | 7992 | 4070 |
| ac1b11: 90-965 |] | | | | 3650 aug | 20 | | 56231 |
| acibility 90-965 rev | | | 344AT-81-09 13 FBB 81 | 13 MK S | 4 Man Po 6 JW81 | WELD TIG | 7962 | 4071 |
| N | | | | 160415 | 308 Days | 2, | | S # 230 |
| | | | 344/AT-81-086 | 4 may 81 | 4 Man 80 | 174PA/17-4ACRF16 WELD TIG | 7991 | 4072 |
| 1 1 | 1 | | | ٧٠٠ | 36< Days | 2, | | SE 231 |
| | 1 | 8803-947047, rev. A | on: Report No. | ospace Textr | 1: Bell Aer | *Specification | | |

| 9 11 | UMBER | TES | TPCT | DURATION | | | | | | |
|-------------|-------|-----------------------------|----------------------|--------------------|-----------------------|-------------------------------|-----------|------------|--------------|-----|
| SPECIME | 1 302 | MATERIAL MON DESCRIPTION | CELL | REPRIG- ERATION | ANALYSIS DOCUMENT, | REMARKS | | | TOTH | 1* |
| BAT No | | CPR NA | IN/OUT | IN/OUT | IOM, etc. | | | | 145 | 1 4 |
| 4073 | 7993 | INCONEL 902 NISPAN C | 6 4 MAI 80 | CZAN | DATE DATE | - | | \ | 90 | • |
| ł . | 1 | TUBE | CUAN 81 | IN MALAA | 13 FEB 81 | special capeul mech 1.856 dia | 7 | i | 1 1 2 | h |
| 50 240 | | 22 | 300 | 1 | , | ł | 1 | Ì | 1 | i |
| 9074 | 7994 | INCONEL 902 | & Han 80 | 10 DAYS | 10. 10. 10.01 | | 1 | ŀ | | ı |
| l | | NI SPANC TUBE | 4 Aren 81 | 26 Mar 81 | 12 WAY & | special capeul met 0.856 dia | 1 | | | i |
| SC 241 | | 22 | 12,50 | 22 | , , , , , | | 1 | | | 1 |
| 4075 | 7984 | 347 CRES | 3650m | 4400 80 | 7.4. | | 1 1 | | | |
| l l | 1 | V64 | 4400 80 | 20400 \$0 | 714 AT . 80 173 | | | ğ | ē | |
| PA 250 | 1 | 23 | 1 245 Days | | | | 1 | (BTAG) | | 1 |
| 4076 | 7983 | 347 CRES 6 | 1 | 16 12443 | | | Hydrazine | | | |
| | 1 | V64 | 9 Mar 80 4 Amery | 4 man 81 | 344AT - 81-014 | | , i | - 6 | 1970 | |
| 81 251 | } | | | 20Apr 81 | 15 MAY &1 | | ğ | 3 | ۾ پ | |
| 4017 | 7995 | 347 CRES 6 | 365ham | 47 | | 1 | 흵 | 2 | | 5 |
| 70// | // 43 | ANNEALED | 4 May 80 | 4400 80 | 244 AT - (x0.17) | | 8 | PROJECTI L | 3 3 | - |
| BA 260 | | TUBE | 245 Days | 1 8400 80 | 2045 0 | | | | ا کا | - |
| 4078 | 7001 | 24 | 9] | 14 D445 | 1 | Į. | | Ë | - # | |
| 7010 | 7996 | 347 CRES 6 ANNEALED | 4 Mar 80 4 Mar 81 | | 344 AT. 81-086 | | rroposa! | = | - 1 | ~ |
| BA 261 | | TUBE | ľ | 24/g 81 | 15 MAY 81 | 19 | | 8 | 4 | |
| | 100- | 24 | 365Days | | 1 | 1 | 8 | 3 | 1 | : |
| 4079 | | 397/347 6 WELD ASTROARC | | 4 NW 80 | 344 AT 30 173 | | 8 | patibilaty | K | _ |
| 84 270 | i | # 78 | , , , , | 14400 80 | 2 UEC 80 | 1 | S Tev | 5 | 3 | |
| | | 25 | 241 :0 1.43 | 15 DA15 | ļ | 1 | 13 | ٦) | 112 | |
| 4080 | | 347/347 6 | 4 MAY 80 L | IANCE SI | SULAT-XI-ON | | ~ | -1 | | |
| R4 2- | | 4 | 7,744.1 | 12/19/25 | 18 MAYSI | 1 | | | 14 | , |
| 84 271 | | # 78 | 36 (Day | | 1 | } | | 1 | | |
| | | *Specification | Rell Asses | | | 903-947047, rev. A | ı | 1 | M | |

| BAT No. | CAPSULE | MATERIAL MONTHS DESCRIPTION 847 CPR No. EPR 5/5 SEAL PARKER KRYTOX 240AC | IN/OUT # Man #0 /0 APR 80 | DURATION REFRIGERATION IN/OUT IN APR SO | ANALYSIS DOCUMENT, IOM, etc. DATE 344A7-80-076 7 May 1980 | REMARKS Terminate - JPL neport 90: 965-9 | | (A) | REVISION TOTH |
|---------|---------|---|--|--|---|--|-----------------------|--------------|---------------|
| | 1990 | EPR 515 6 SEAL PARKER KRYTOX 240AC | 37 days 4 Mar 80 8 May 80 65 days | 8 MAY 80 | 344 AT-80-09/ 28 MAY 1980 | (A) Terminate – JPL report 90:965-9 (A) | | | |
| | | | | | | | Hydrazine, I | | July 1979 |
| | | | | | | | Monopropellant Grades | JPL Proposal | 79X07500 |
| | | | | | | | de. | 90-965 rev. | |
| | | | | | | | | 2 | |

| NUM TEST BAT No. | BER CAPSULE | MATERIAL MONT DESCRIPTION SAT | | REFRIG- ERATION IN/OUT | ANALYSIS DOCUMENT, IOM, etc. DATE | REMARKS | The state of the s | REVISION | TOT! |
|------------------------|----------------|-------------------------------------|---|---------------------------------|-----------------------------------|---------------------|--|--------------------------------|-------------|
| 4/00 | 7949 | No Ha only 2 | 4 28 Jan 80 4 5897 80 | 45B743 | 7/100 80 | control | | | C 3 |
| 4101 | 7908 | 40cc NeHe only 2 | 2 FHAN 80 4 FHAN 80 | 34 0445 4 May 81 30 Mp 81 | 3447-81-086 15448 81 | control | | | |
| 4102 | 79/6 | 40 acc No Ha soly 2 | 365 Bang 8 8 Mar 80 4 Sept 81 | 4 SEP 81 8 FL8 82 | энл 82/17 13 Мо/82 | control | Bydraziı | (BAVE) | |
| 4103 | 7904 | No. He Douby 2 | 549 1 28 Jan 80 4507 30 220 Dags | M OCT 40 | 744 AT. 90-160 | control 60°C | ne, Monopro | PROJECT | 1979 793075 |
| 4104 | 7931 | No. Ha and is | | 4 man 81 25 Man 81 | 344 /ET -81-DM | control 60°C | pellant or | Materia JPL Pro | 8 |
| 4105 | 7942 | | | 28 JUL 81 10 FEB 82 198 | 344AT-82-117 13 MA-, 82 | control 60°C | rade | rial Compatib Proposal 90-9 | |
| 4106 | 7985 | Na Ha only 2 | 4 4 Mar 80 | 3 MAR 82 31 MAR 82 | 344AT-82-11'1 13 Mb., 42 | control | | etibility 90-965 rev. 2 | |
| 4107 | 7910 | 40 ex No Ha only 2 | # HAN 80 USMEN'NZ | 3 MAR 82 24 NINH 82 | 344AT 82 117 | control 60°C | | | |
| | <u></u> | 40 KK | /// a | 22 | <u> </u> | 8803-947047, rev. A | { | | |

| CAPSULE | MATERIAL MAN | 7/80 | | | | | | |
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| | DESCRIPTION | CELL | REFRIG- ERATION | DOCUMENT, IOM, etc. | REMARKS | BAISION | 91 | ٧ |
| | BAT COR No. | IN/OUT | IN/OUT | DATE | | " | 33 | |
| | | | · · | 344A1-82-117 | control | | | |
| j | 40 ccc | 77.9 | 7 | | | | | |
| 79/3 | N2H4 only 3 | 4 FMAR | | | control | | | |
| , ,,, | , | | | 13 May 42 | 60°C | | | |
| 7917 | | | | + | ralibration unit | A 5 | , ĝ | İ |
| //0/ | | | | | open funnel | 164 | P 1 | |
| 70.10 | Can. + | 14 10 Man | 80 | | califiation unit | | 979 | |
| 7977 | Emgay - | | | | open funnel | PROJECT | 79X075 | |
| 7905 | J-440-5-4 | | PO | | Reference unit | JPL JPL 11an | 8 8 | |
| 7997 | Sealed & Enternal pressure 733 mm hz | 21 18 Man | 80 | | Reference unit | Compatib cosal 90-9 | | |
| NONE Nogauge | No Ha only | 8 apr 8 | 80 8 apr 80 | May 1980 | Special for CO2 analysis check Terminated | 111ty 65 rev. 2 | | mas 11 |
| 79201 | Empty : | 2 \$ 10 Mar | 80 | | califration unit | | | 8 |
| | 7919 7913 7907 7917 7905 NONE Nogauge | 7919 112 's only 40 CK 7913 N2H4 only 40 CK 7907 Empty 2 7907 Empty 2 7905 Sealed preson 733 mm lig 7997 Enternal preson 733 mm lig NONE N2 H4 only Nogenge 40 CK | 7919 112 4 ANDY 24 9 MAR 03 Man 40 CK 17.9 7913 N2H4 only 24 9 MAR 03 Man 40 CK 77.9 7907 Empty 24 10 Man 79.9 7917 Empty 24 10 Man 24 18 Man 25 minus pressure 733 mm hg 7997 Sealed 21 18 Man 233 mm hg 7997 Sealed 21 18 Man 1833 mm hg NONE N2 H4 only - 38 Fet 8 april 40 CK 40 day | 7919 112 4 May 24 9 MAR 80 03 Man 82 17.9 7913 N2 H4 only 24 9 MAR 80 24 MAR 82 24 MAR 82 24 MAR 82 24 MAR 82 27.9 22 7907 Empty 28 10 Man 80 22 7907 Empty 24 10 Man 80 22 7907 Empty 24 10 Man 80 22 7907 Empty 24 18 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 20 Man 80 20 Ma | 7919 112 4 Anly 24 AMAR80 3 MAR 82 344AT-82-117 13 May 82 7913 N2H4 only 24 AMAR80 3 MAR 82 344AT 82-117 13 May 92 40 CC 779 22 7907 Empty 24 10 Mar 80 7905 Sealed 24 18 Mar 80 Internal pressure 733 mm hg 7997 Sealed 24 18 Mar 80 Internal pressure 733 mm hg NONE N2 H4 only - 88 Fet 80 20 Fet 80 344AT-80-08 8 apr 80 8 apr 80 May 1980 40 CC 40 days \$0 days | 7919 112 14 only 24 4 MAR80 03 MAR 82 2144 182-117 Control 7913 N2H4 only 24 14 MAR80 3 MAR 82 2144 182-117 Control 93 May 92 60°C 7907 Empty 24 10 Mar 80 Calibration unit 00°C 7905 Sealed 24 18 Mar 80 Reference unit 24 MAR 80 Reference unit 25 mm hg 7997 Sealed 24 18 Mar 80 Reference unit 100°C 17905 Sealed 24 18 Mar 80 Reference unit 1733 mm hg 18 Mar 80 Reference unit | 7919 112 4 only 24 4 MAR80 03 MAR 82 214 17 13 May 82 117 13 May 82 117 Control 60°C 7913 N2 H4 only 24 4 MAR80 3 MAR 82 24 MAY 82 117 13 May 92 60°C 7907 Empty 24 10 Mar 80 calibration unit open funnel frame of 60°C 7917 Empty 24 10 Mar 80 calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit open funnel for calibration unit for calibration unit open funnel for calibration unit for calibration | 7919 112 4 ANAR 80 03 AMAR 80 179 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 13 May 92 Control 14 Tol 14 |

| REVISION | | REMARKS | ANALYSIS DOCUMENT, IOM, etc. DATE | URATION REPRIG- ERATION | CELL IN/OUT | MATERIAL DESCRIPTION BAT CPR No. | CAPSULE | NUM TEST Bat No. |
|---|-----------------------|--|-----------------------------------|--------------------------|----------------|----------------------------------|---------|------------------------|
| | | colitation unit open funnel 60°C | | | 18 Mar FI | BAT CPR No. Empty 14 | 79203 | 4116 |
| OATE) | Bydrazine, | | | | | | | |
| | Monoprop | | | | | | | |
| Material Com JPL Proposal | Monopropellant Grade* | | | | | | | |
| Material Compatibility JPL Proposal 90-965 rev. | de. | | | | | | | |
| lity 5 rev. 2 | | | | | | | | |
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| | 1 | . 8803-947047, rev. A | on; Report No. | nspace Texts | n: Bell Aer | *Specification | 1 | |

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| | | | PULSION LABORATORY INSTITUTE OF TECHNOLOGY | |
| | 3 | SUMMAR | Y | |
| | TEST | CAPSULE , | SPECIMEN NUMBERS | |
| | DETA | IL INFO | RMATION | |
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| Prep | ared by | TOTH | Dore | |
| | _C. / | YORAN | Daro _ Tuly 1999 | |
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| | · ——— | | ENDIX OR REVISION | |
| Date | Pages Affected | Appendix OR Revision | Remarks | Changed by |
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B-17

JPL 0000 MAN (

Classification

| | NUMBER | | | | | | |
|--------------|----------|--|----------------------|-----------------------------------|------------------------------------|------------------------------------|--|
| SPECIMO | Vol. CAM | DESCRIPTION | TES | T DURATION SEACEO REFRIG- ERATION | ANALYSIS DOCUMENT, IOM, etc. | STOP 110% REMARKS UNNEL 110% | |
| NONE | 7901 | Voc. cm ³ Capsule No caps | broke. | EN/OUT | 1 | N2HAVOL = 40.0-Spec Broken 5.10 | WENTED AND LANGE OF THE PARTY O |
| 4006 | 7902 | No caps | 28 Jan 8 | 2174.00 | | | |
| 006 | 88.8 | 0.23// | 6:30 AM | 12N 21°C | | 60°C 6.80 | MORAN |
| 4001 | 1 | | 28 Jan 81 8:30 AM | 2 2 2 20 | | 39.7689 5.40 | |
| 4/03 | 7904 | 0.2320 NoHa 40cc | 28 Jan 80 | 2100 | ENTERATED | 39.768 | (DATE) July 1979 (DAYE) |
| NONE | 86.3 | 0 | 2 20 days | 1 PM 22 °C | to I me me may | 1 -0 1004 1 | K |
| 4/12 None | 7905 | NOTHING PRESSURE: JPL I ATMOSPHERE! | 10.11 | 11 Man 80 10:30 AM | - 1706 | 3.82 2.70 | PROJECTI MATE |
| NONE | 7906 | Experimental | | 23°C | | Ref no ag reading | 79XL |
| 4110 | 7907 | Noture | 40 14 0 5 | | | and and washing | |
| | | NOTHING Open merse | 8:30 AM | | | 4.14 | 7.50/ 90-965 rev |
| 1 | 7908 | N2 H4 40KK | 4 MAR 80 8:30 AM | 27 Feb 80 9:30 AM | | Blank calibration | |
| NONE | 85.6 | O *Specification | | 1200 | | , | |

ORIGINAL PAGE 18
OF POOR QUALITY

| NUM SPECIMEN | BER CAPSULE | MATERIAL | CELL CELL | SEFILED REFRIG- | ANALYSIS DOCUMENT. | REMARKS | PARPAGE 70 REVISION |
|-----------------|----------------|--------------------------------|----------------------|----------------------|--|----------------------------------|--|
| BA | | DESCRIPTION VH am ³ | | ERATION | IOM, etc. | N. Ha and | 707 707 |
| NONE | 7909 | Experimental | IN/OUT | 2N/0117 | DATE | NeHa wol Ref. unit for sag | 1 H/ H/ |
| 4107 | 7910 | N2 H4 40CC | 4 Mar 80 8130 AM | 17 Feb 80 9:30 AM | ······································ | 60°C 3.54 Control 4.80 | MORAN |
| NONE | 85.8 | 0 | | 23°C | | 40.00 | |
| 4023 | 7911 | | | 225an80 10AH | | 60°C 7.13 | (OATE) July IDATE) Bydra: |
| 047 | 87.2 | 0.2698 | | 20°C | | 39.7302 | 197 |
| 4011 | 79/2 | | 28 Jan 80 8:30 AM | 22 Jam80 10 AM | | 60°C: 7.59 Total Not. 42.0 ex | (REPORT NO 9 79X07500 (PROJECT) |
| 016 | 87.9 | 3.3606 | <u> </u> | 20°C | | 36.6394 | DAT N |
| 4109 | 7913 | NeH4 40KK | 4Mar 80 8:30 AM | 27Fet80 9;30 AM | | | |
| NONE | 86.0 | 0 | | 23°C | | 40.00 | rial Com Proposal |
| 4037 | 7914 | | 28 Jan 80 8:30 AM | 18 Jan 80 1 PM | | 4.18 | 2 PAGE terial Compatibility L Proposal 90-965 ray ant Grije* |
| 300 | 87.1 | 0.6657 | | 22°C | | 39.3343 | 1151 |
| 4055 | 7915 | | 285am 80 8:30 AM | 18 Jan 80 11 AH | | Total.vol. 42.0 KZ | PAGE 1sty 5 zav. |
| 118 | 87.1 | 0.7679 | | 2100 | | 39.2321 | ~ 2 |
| 4102 | 7916 | N2H4 40KL | 9 May 80 8:30 AM | 27 Feb 80 10 AM | | Control 8.90 | g |
| NONE | 84.8 | 0 | | 2300 | | 40.00 | |

| num Specimen | BER CAPSULE | MATERIAL DESCRIPTION | TEST E | URATION SEALED REFRIG- | ANALYSIS DOCUMENT, | REMARKS | | NATE. | REVIS | PREPA |
|-----------------|----------------|-------------------------|-----------------------|------------------------------|--------------------|-----------------|--------------|--------|----------------|------------|
| BA | Volem? | Volam? | IN/OUT | ERATION IN COLUMN | IOM, etc. DATE | N2H& NOP | | | NOI | 75 |
| 4111 | 7917 | NOTHING | 10 MAR 80 8: 30 AM | | | 60°C | 8.36 7.50 |] | , | #// |
| | | Open nech | | i | | Blank | | | | YOU |
| 4041 | 7918 | | 28 Jan 80 8: 30 AM | 18 Jan 80 | | 60°C. | 4.91 | | | PAN |
| 304 | 87.0 | 0.6492 | | 22°c | | 39.3508 | <u> </u> |] | | |
| 4/08 | 1919 | N2 H4 40 KK | 4 Mar 80 8; 30 AM | 27 Feb. 80 :0 AM | | Control | 8.2 | Hydra2 | DATE) | July |
| NONE | 84.8 | 0 | | 23°C | | 40.00 | | ing. | 1 | 1979 |
| 4046 | 7920 | | 4 May 80 8: 30 AM | 27 Fel 80 10 30 AM | | | 5.3 | Mono | CORF. | 79X |
| 101 | 86.8 | 1.0618 | | 23.0 | | 38.938 | | prop | 3 |)750 |
| 4005 | 7921 | | 28 Jan 80 8:30 AM | 21 JAM 80 12 N | | 60°C. | 7.0 | ellant | Mater JPL P | æ 6 |
| 005 | 88.6 | 0.2367 | | 21°C | | 39.7633 | | न्त | 19 67 | |
| 4027 | 7922 | | 18 Mar 80 9:00 AH | 11 Mar80 1:30 | | 60°C | 8.10 | de* | Compat | |
| 058 | 85.3 | 1.5605 | | 23.0 | 21 | 38.4395 | | 1 | ib11 -965 | |
| 4056 | 7923 | | 28 Jan 80 8:31 AM | 215Am 50 3PM | | Total not. 42.0 | 5.90 EC | | 10-965 rev. | |
| 119 | 88.7 | 0.7494 | | 21°C | | 39.2506 | | 1 | 2 | |
| 4002 | 7924 | | 28 Jan 80 8:30 AM | 21 Jan 60 3 PM | | | 5.35 | | | |
| 002 | 85.1 | 0.2371 | ì | 31ºC | | 39.7629 | | | | |

| NUM | | MATERIAL | CELL CELL | REFRIG- | ANALYSIS DOCUMENT, IOM, etc. | REMARKS | | TOT TOTAL |
|---------|-----------|-------------|-----------------|-------------|------------------------------------|-----------------------|---------------|--|
| PECIMEN | CAPSULE | DESCRIPTION | | ERATION | DATE | N2 H9 Vol | 5.85 | 7 7 3 |
| BA | Val and | Volan? | IN/OUT | 21Jan80 | | 60°C | 3.03 | |
| | 7925 | | 8:30 AM | ILAM | | | 1 | |
| 4019 | / / | | | 21.0 | | 39.917 | 5.55 | MORAN |
| | 90.5 | 0.083 | 30 Tan 80 | 215 m80 | | | | اتخا |
| 036 | 70 16 | | grao AM | 4PM | 1 | | 1 | |
| 4017 | 11,00 | | | 21°C | | 39.916 | 5.1 | DATE (DATE |
| 034 | 90.2 | 0.084 | 2016 6 | 0 21 Jan 80 | | 60°C | 5.1 Bydrazine | (DATE) (NEP JULY 1973 79) (DATE) (PRO |
| | 72 27 | | 28 Jan 8 | | 1 | | | 1973 |
| 4042 | 17-1 | 1 | | 210 | | 39,3294 | 100 | |
| 11 | 87.4 | 0.6706 | 28 Jan 8 | | 1 | 60°C | 6,73 | (REPORT NO. 19X07500 |
| 305 | 7028 | | 9:30 AM | 10 AM | | | | 1 2 3 3 |
| 4024 | 17720 | 1 | 1 | 20.0 | | 39. 7347 | | 2 20 |
| 1 | 83.1 | 0.2653 | 28 Jan 8 | | 0 | | 3.7 | ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) ONT NO.) |
| 048 | 120 76 | 1 | 8:30 A | 4 3PM | | | | Haterial Company PL Proposal S |
| 2 4003 | 3 7929 | | 1 | 21.0 | | 39.7655 | 9.25 | lal Comproposal |
| 8 | 84.2 | 0.2345 | | | 20 | 60°C. | 7. 23 | |
| 003 | | | 285mg 8:70 A | M 3PM | ١, | | , | |
| 3 404 | 4 7930 | | | 2100 | 1 | 39.364 | 9.5 | |
| N . | 1000 | 0.6360 | CC 285m | | 0 | 60°C. | 4.5 | PAGE_ paribility 90-965 rev |
| 30 | 1 1 2 2 2 | 11 11. 40. | CC 2 30 A | MIPM | " | | | 4 4 |
| 1410 | 4 1773 | | 1 | 1,,,, | 1 | 40.00 | 6.75 | |
| * A/a | VE 88. | 0 | | 80 21Jan | FC | 60°C. | 9.73 | |
| No | | | 28 Jan 8:30 | AIT O PM | · | | | 1 1 1 11 |
| 2 40 | 07 793 | | 1 | | | 39.7676 | | |
| 2 00 | 100 | 2 0.2324 | | | Repor | t No. 8803-947047, re | v. A | |

| NUM SPECIMEN | BER CAPSULE | MATERIAL | TEST | DURATION SERIED REFRIG- | ANALYSIS DOCUMENT. | REMARI | ts | REVI |
|-----------------|-------------|-------------|-----------------------|-------------------------------|-----------------------|-------------------|------|--------------------------------|
| BA | Vol an | Volum 3 | | E. TION | IOM, etc. DATE | NeHand | _ | TO 7 |
| 4018 | 79 33 | V-170m2 | 28 Jan 80 8.3000 | 21 Jan 80 4 PM | DATE | 1 1 2 1 4 N 3 1 | 5.50 | H // |
| 035 | 91.9 | 0.086 | | 2.1°C | | 39.914 | | 40 |
| 4053 | 7934 | | 4 May 80 8:30 AM | 27F24.80 | | | 5.90 | MORAL |
| 115 | 86.1 | 0.28/5 | | 23°C | | 39.7185 | | |
| 4020 | 7935 | | 28JAM80 8:30 AM | 21Jan80 4PM | | 60°C | 5.85 | (DATE) July (DATE) Hydras |
| 037 | 87.6 | 0.087 | | 2100 | | 39.913 | | 1979 |
| 4008 | 7936 | | 28 Jan 80 8:30 AM | 21Jan 80 2 PM | | 60°C | 6.75 | 9 79X0 |
| 008 | 85.4 | 0.2301 | | 2100 | | 39.7699 | | 075 075 08671 |
| 4009 | 7937 | | 28 Jan 80 | 21 Jan 80 11 AM | | | 6.0 | Mate: JPL 1 |
| 014 | 36.2 | 2,2708 | | 2:00 | | 37. 7292 | | er fig |
| 4012 | 7938 | | 28 Jan 80 8; 30 AM | 21 Jan 80 | | 60°C | 7.9 | rial Compa Proposal S |
| 017 | | 2.5323 | | 21.0 | | 37.4677 | | 3 11 |
| 4032 | 7939 | | 18 Mar 80 9:00 AM | 11 Mar 80 1:30 PM | | 60°C | 7.85 | Compatibility Deal 90-965 rev. |
| 670 | 86.2 | 1,4709 | | 23°c | 24 | 38,5291 | | N |
| 4021 | 79 40 | | 28 Jan 80 8:30 AM | 23 JA 1150 | | | 5.65 | |
| 045 | 88.3 | 0.2620 | | 20°C | | 39.738 | | |
| L | | *Specificat | ion: Bell Aer | ospace Textro | n; Report Mo. | 8803-947047, rev. | A | |

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| l | | | P/ 30 44 | IPM | | | 3.92 | |
| 120 | 83.8 | 0.7366 | | 22.0 | | | | |
| 4105 | 79 42 | N2H4 FORK | 28 Jan 80 | 18 Jan 80 | | 39.2634 | | |
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| NONE | 88.3 | 0 | 1 | 22°C | | Control | | AR |
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| | 88.1 | 0,6430 | 1 | 21.0 | | 20- | | (DATE) |
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| 046 | 00 - | | 8:3nAM | 10 AM | | | 6.68 | |
| | 84.2 | 0.2676 | 1 1 | 20°c | | 20 | | 3 2 3 |
| 4038 | 945 | | 28 Jan 60 | 18 Jan 80 | | 39.7324 | 2,0 | (REPORT NO.) 79X07500 (PROJECT) N. |
| 301 | 82.7 | | 8:30 AM | IPM | | 1 | ¥ · O | 14 1 S 5 |
| | 946 | 1,6299 | | 22°C | | 20 | in a | |
| 702// | 770 | | 9 May 80 3 | 17 Feb 80 | | 39.3701 | 3 | rial Com |
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| 1004 7 | 947 | | 1071 | 23.0 | 24 | 38,6652 | - 1 | 98 |
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| 104 8 | 6.8 0 | 2363 | | 3 PM | | | 5,88 | 25 2 |
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| 302 | 86.8 | 0,5506 | | 2100 | | 39.4494 | ì | 8 | Ē | 7 |
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| 303 | 85.3 | 0.6097 | | 22°C | | 39.3903 | - | | | |
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| 057 | 83.5 | 1.5745 | | 2:05 PM 23°C | 29 | 38.4255 | | orgon | 79X075 |
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| 4 1 | 80.9 | 1.3813 | | 23°6 | 24 | 38.6187 | | B (| |
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| 127 | 85.3 | 0.0056 | | 2300 | 24 | 39,9944 | Ì | | |
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| 068 86.1 1.4278 | | 2300 | • | 1 30 500 | | |
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| 4033 7969 | 4 Mar 30 | 27 Fel 80 | | 39,9939 | | PROJECTI MA |
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| | SE 201 | 84.1 | 1.5179 | | 2300 | | 38.4821 | 5.6 | MOR | |
| 14 | 4051 | 7974 | | 4 Mar 80 8: 30 A M | 27 Feb 80 2.95 PM | | | 3,6 | JAN | |
| | 112 | 83.3 | 0.5820 | | 23.0 | | 39.418 | 5,4 | | 1 |
| 6 | 4063 | 7975 | | \$ May \$0 8:30 AM | 27 Feb 80 10:30 AM | | | 3,7 | DATE) DATE Bydrazi | |
| Ì | 133 | 86.7 | 0.1157 | | 23°C | | 39.8843 | | 1979 (ne. | 1 |
| 4 | 4064 | 7976 | | 4 Mar 80 8:30 AM | 27 Fet 80 11 AM | | | 5.1 | 7910 7910 89004 | |
| ٦ | 134 | 87.5 | 0.1191 | ļ | \$3°C | | 39,8809 | | | |
| 14 | 4052 | | | 4 Mar 60 8: 30 AM | 27 Feb 80 2:45 PM | | | 4.24 | Haterd JPL Pr | |
| | 113 | 83.9 | 0,5916 | | 2300 | | 39.4084 | | 2 8 F | } |
| 14 | 4034 | 7978 | | 4 Mar 80 8:30 AM | 27F2480 2;95PM | | | 6.2 | Compat eal 90 | |
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| 4050 | 7981 | | 4 Mar 80 8:30 AM | 27 Feb. 80 3 PM | | | 4.20 | |
| 110 | 87.3 | 1.8275 | | 23.0 | | 38.1725 | 1 | 1 2 |
| 4045 | | | 4 May 80 8: 30 AM | 27 Fel 50 10 AM | | | 4.70 | MORAN |
| 100 | 87.2 | 1.0567 | 1 | 23% | | 38,9433 | j | |
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| 251 | 85.5 | 1.6193 | | 23.0 | | 38.3807 | | 1979 |
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| 250 | 87.6 | 1.6191 | | 33.0 | | 38,3809 | [| |
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| 270 | 85.8 | 0.8548 | } | 2300 | | 39.1452 | | . 2 |
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| 280 | 87.8 | 0,4024 | 37 dys | 23.0 | | 39.5976 | | |
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| 4080 | 7989 | ! | 9:30 AM | 3PM | | 1 | 3,70 | | |
| 271 | 85.2 | 0.8496 | | 27.0 | | 39.1504 | 4, 2 | | MORAL |
| 4082 | 7990 | EPR 515 Oring Kry tox 240AC | 4 May 80 8:30 AM 8 May 80 | 27 Feb 50 2:10 FM | | W/A 4102, 4103 | 7, 4 | | 241 |
| 281 | 86.6 | 0.4118 | 65 dys 10:3 | | | 39,5882 | 4.5 | _ | B 5 |
| 4072 | 7991 | | 4 Mar 80 81 30 AM | 27Fef 80 1 PM | | | 7,3 | Bydrazin | IDATE) _ July IDATE) |
| SE 231 | 90.3 | 1.5383 | | 23.0 | | 38.4617 | | ine | 1979 |
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| 50240 | 87.2 | 0.8975 | | יינפ יי | | 39.1025 | | S. | riel Topo |
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| 260 | 87.5 | 0. 9202 | | 2300 | | 39.0799 | | | 2 |
| 4078 | 7996 | | 4 Mar 80 8: 30 AM | 27 Fel-81 | | | 3.6 | | |
| 261 | 86.7 | 0.8960 | | 3 PM 23°C | | 39.104 | | | 1 1 1 |

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| 014 | 103.6 | 1.3765 | | | ↓ | 60°C | 7 | |
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| 02 | 4 (02) | 1.3479 | | | | 60°C | - N | [S] |
| 4016 | 79206 | | 18 May 80 9:00 AM | | | 38.6393 | | rial Compatibility Proposal 90-965 rev. |
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APPENDIX C

CO2 ANALYSIS

A. DETERMINATION OF CARBON DIOXIDE ABSORBED BY HYDRAZINE

The general laboratory test setup for CO_2 analysis is shown schematically in Fig. C-1. The sulfamic acid solution is prepared by dissolving 150 g of reagent grade material in 1.0 liter of distilled water. To reduce the CO_2 content of the sulfamic acid, high-purity helium, passed through Ascarite, is bubbled through the sulfamic acid solution via the glass frit, which provides a fine gas dispersion and efficient purging. The helium gas is passed through the sulfamic acid delivery tube for about 16 hours at 50-60 cm³/min. The exit end of the helium gas from the sulfamic acid bottle is protected against air and CO_2 with an Ascarite tube. This Ascarite tube is replaced with a new one after the helium purge. With the precautions outlined, the blank CO_2 is under 2.0 ppm.

The apparatus is standardized by means of a NaHCO3 solution prepared by dissolving 0.381 g of dried NaHCO3 in 1.0 liter of distilled water. The solution is stored in glass, and air exposure is minimized. This solution vides 0.20 mg CO2 per milliliter. Its CO2 content is 200 ppm by weight.

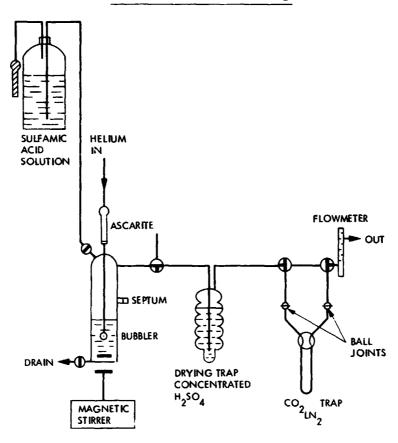
The column is 6.0-mm-diam tubing, 3.66 m long (0.24-in.-diam, 12 ft long), filled with 60 to 80 mesh F & M Polypack No. 5. This packing gives good separation of CO_2 at ambient temperature. The peaks are sharp, permitting direct reading of the heights and eliminating the need for peak area measurements. The column is bent into a number of 0.7-m (2-ft) sections arranged close together and contained in a glass jacket. The filament-type thermal conductivity detector unit is kept at ambient temperature in a glass dewar to minimize temperature fluctuations. A 1.0-mV recorder records the detector output. Helium flow is $60 \text{ cm}^3/\text{min}$.

The first step in the analysis is the determination of the blank: the ${\rm CO_2}$ picked up from the reagents and the system. The flow of the high-purity helium purge gas, after passage through Ascarite, is adjusted to $50~{\rm cm}^3/{\rm min}$ by means of a flowmeter in the system. A $60-{\rm ml}$ sulfamic acid solution is run into the unit via the stopcock. The stirrer is adjusted to give vigorous constant stirring. Once set, the helium flow and stirring are kept fixed through the whole run.

After addition of the sulfamic acid, the helium gas is passed through the traps for 30 min to purge the system of air. The $\rm CO_2$ trap is then immersed in liquid nitrogen to the top level of the glass beads. The flow of helium is continued for 20 min, after which time the stopcock on the $\rm CO_2$ trap is turned to isolate the loop on the trap.

The trap, immersed in liquid nitrogen, is transferred to the gas chromatograph sampling system. The stopcock on the CO_2 trap is turned so as to evacuate the noncondensable gases in the trap and then turned to isolate the loop containing the frozen CO_2 .

LIBERATION AND TRAPPING OF CO_2



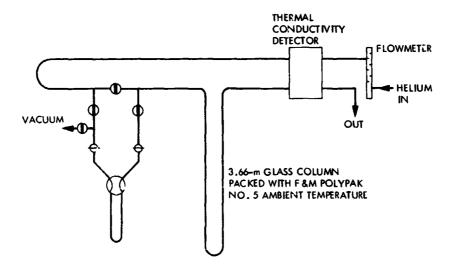


Figure C-1. Test Procedures for ${\rm CO_2}$ Analysis

The next step is to flow the helium gas through the branched leg of the sampling system. During this operation the liquid nitrogen is removed, and the CO_2 trap thawed with warm water. After a few minutes, the stopcock is turned so as to flush the CO_2 with helium into the chromatographic column for separation and assay. The blank run is repeated until consistent, low values are obtained.

The standardization run is made in the same manner as the blanks, except that after a 5-min preliminary purge with helium, 0.50 ml of standard NaHCO $_3$ solution is injected into the vigorously stirred sulfamic acid via the septum on the sulfamic acid unit. The released CO $_2$ is frozen out during the 20-min duration in the CO $_2$ trap immersed in liquid nitrogen. The trapped CO $_2$ from the standard solution is transferred to the gas chromatographic sampling system. This yields a peak height for a standard of 100 ppm CO $_2$.

The CO₂ in the hydrazine is similarly determined. A 1.0-ml sample is injected into the sulfamic acid solution via the septum, and the released CO₂ is swept out of the solution for a period of 20 min. The hydrazine injects should be made rapidly with a minimum exposure to air. The sulfamic according is sufficient to neutralize 1.0 ml of hydrazine and should therefore discarded after each hydrazine analysis. If another sample is to be run, the sulfamic acid unit is refilled, and the blank and the standard determinations are made as before.

B. CALCULATION FOR CARBON DIOXIDE CONTENT

The formula for determining parts per million of carbon dioxide is

For hydrazine, where the density can be taken as 1.0, a density correction term is not applied. The error due to this omission is about 1%, well within the $\pm 10\%$ precision for CO₂ determination when the values are under 20 ppm.

C. CONCLUSION

The method described provides meaningful results for the determination of ${\rm CO}_2$ in hydrazine or its methyl-substituted derivatives.

APPENDIX D

TEST COUPON PHOTOGRAPHS IN THE POSTTEST CONDITION

Figure D-l shows test specimens from Program A, the secondary containment system; Figure D-2 shows test specimens from Program B, the primary containment system.

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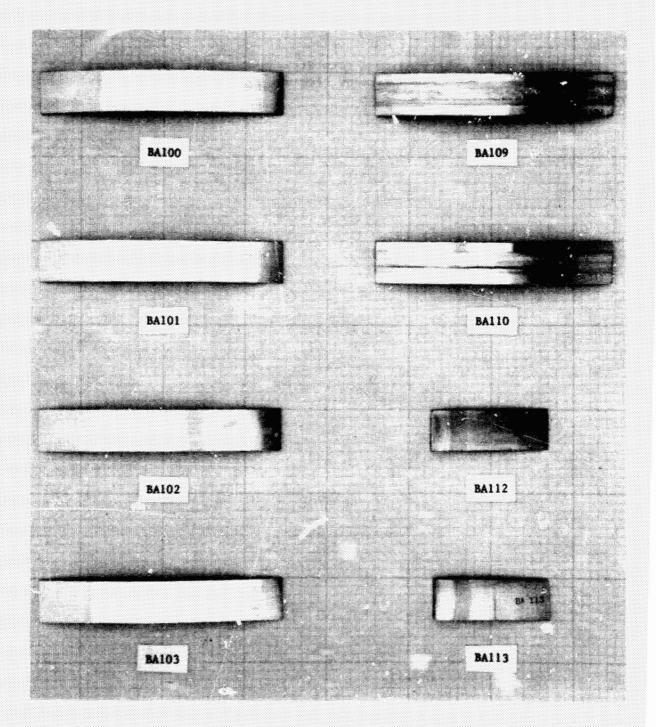


Figure D-1. Test Specimens, Hydrazine Decomposition Program "A" - Secondary Containment System

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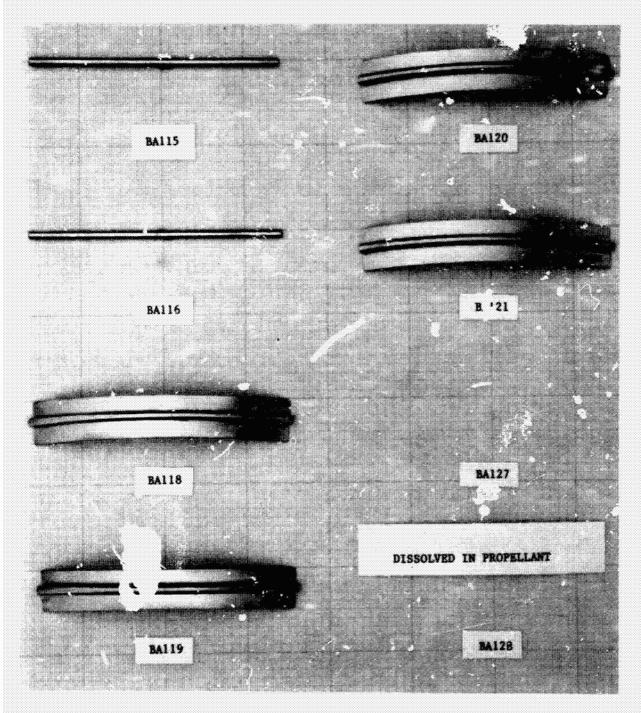


Figure D-1. Test Specimens, Hydrazine Decomposition Program "A" - Secondary Containment System (Continued)

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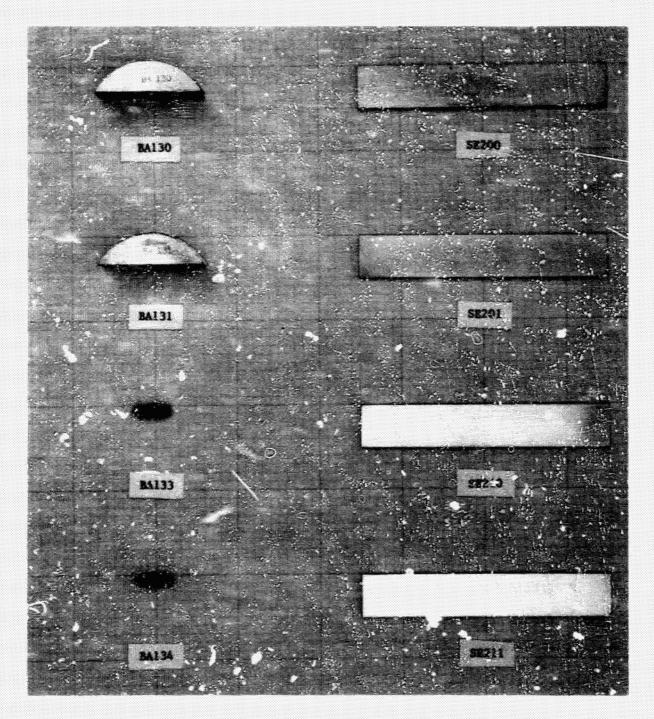


Figure D-1. t Specimens, Hydrazine Decomposition Program "A" - Secondary Containment System (Continued)

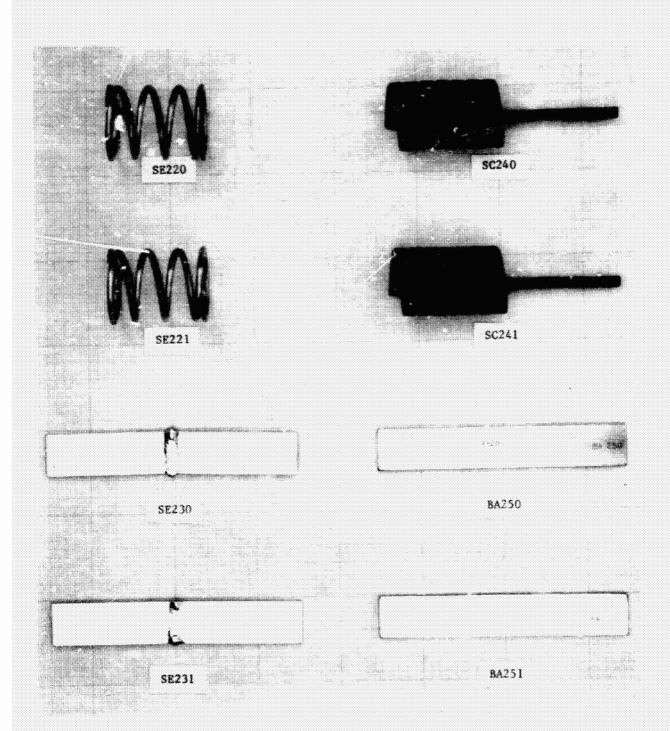


Figure D-1. Test Specimens, Hydrazine Decomposition Program "A" - Secondary Contamment System (Continued)

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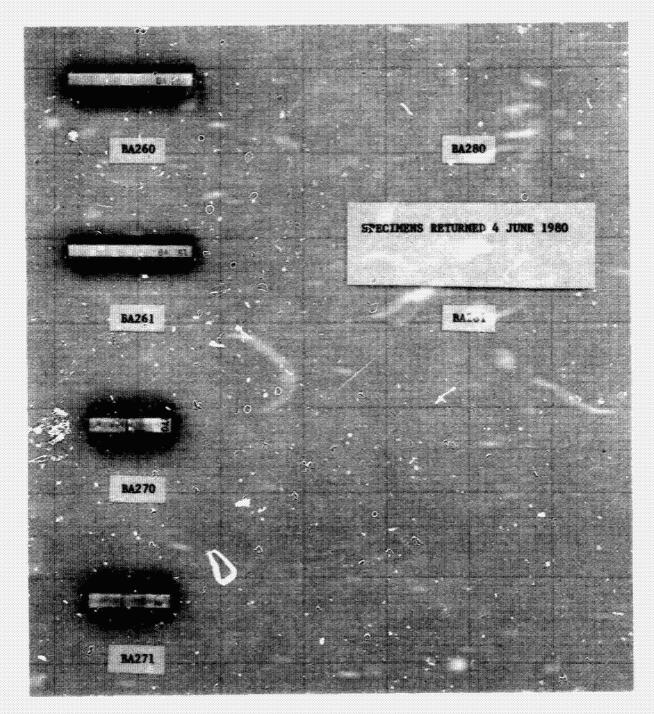
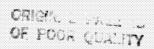


Figure D-1. Test Specimens, Hydrazine Decomposition Program "A" - Secondary Containment System (Concluded)



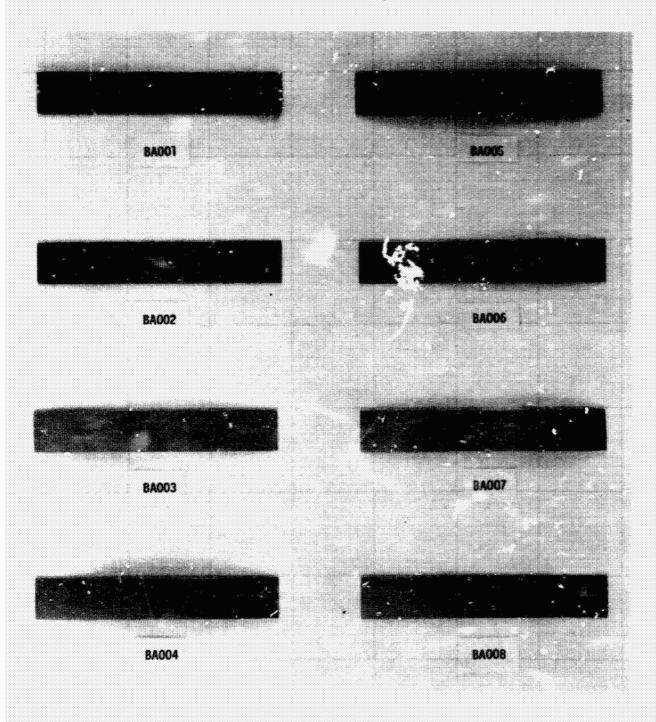


Figure D-2. Test Specimens, Hydrazine Decomposition Program "B" - Primary Containment System

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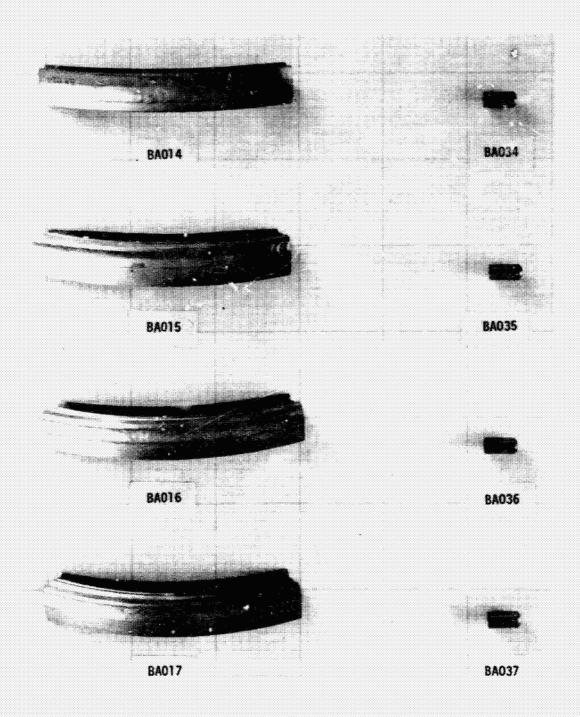


Figure D-2. Test Specimens, Hydrazine Decomposition Program "B" - Primary Containment System (Continued)

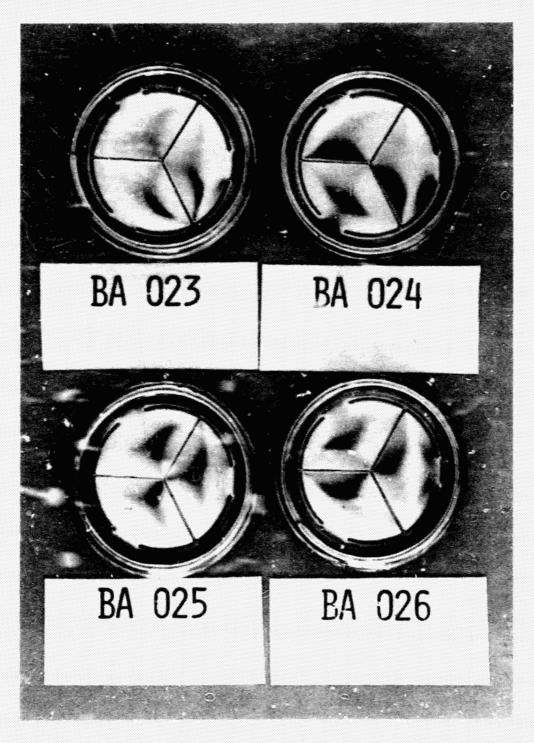


Figure D-2. Test Specimens, Hydrazine Decomposition Program "B" - Primary Containment System (Continued)

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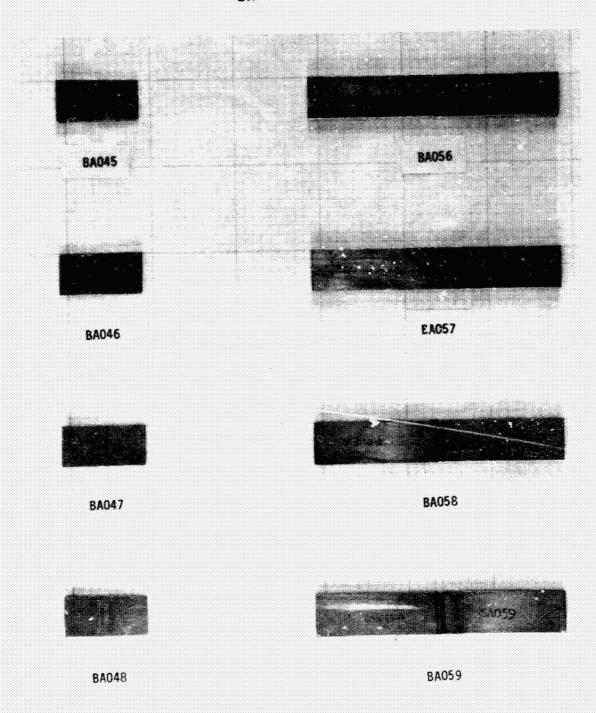


Figure D-2. Test Specimens, Hydrazine Decomposition Program "B" - Primary Containment System (Continued)

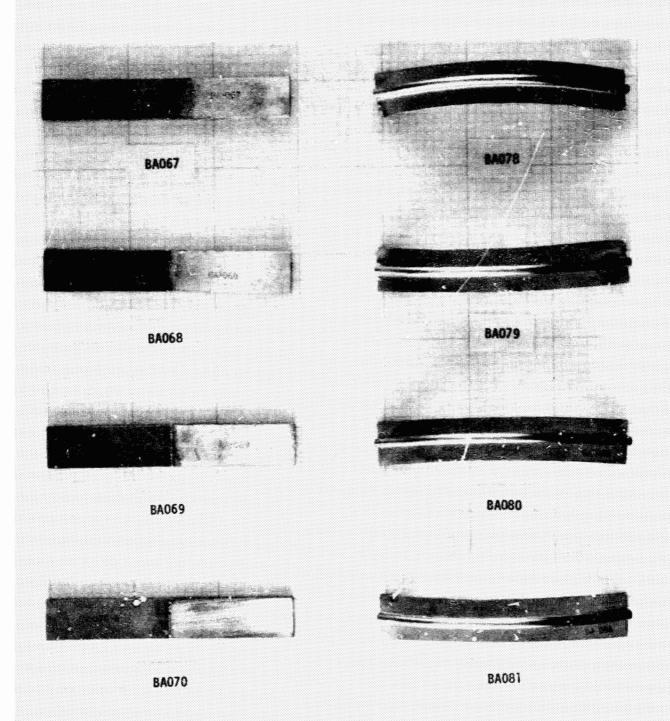


Figure D-2. Test Specimens, Hydrazine Decomposition Program "B" - Primary Containment System (Continued)

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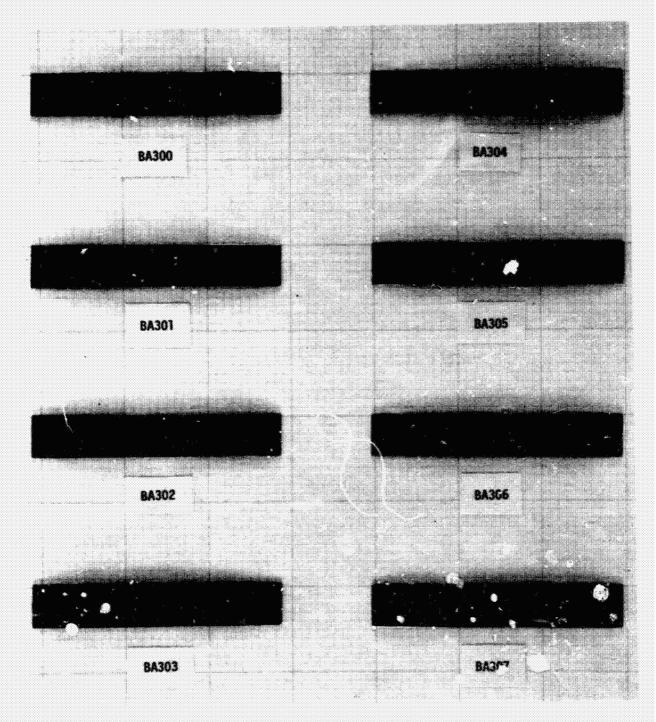


Figure D-2. Test Specimens, Hydrazine Decomposition Program "P" - Primary Containment System (Concluded)

APPENDIX E

347 CRES WELDS

A. INTRODUCTION

Some concern was expressed by the sponsor that the 347 CRES diaphragm in the burst disc assemblies (BA 023 to BA 026) might be subject to stress corrosion cracking in a hydrazine environment. All four specimens were removed from storage at 12.5 months rather than the scheduled 24 months. A cursory examination at JPL was inconclusive. However, a very thorough examination by BAT revealed that any crack formation in the diaphragms was due to the manufacturing and coining (crimping) process.

In the NASA long-term material compatibility test program, a few welded 347 CRES specimens were still in storage. Two were removed - one of which had been stored in the stressed configuration. These were thoroughly tested to determine the differences induced by storage while stressed versus storage while relaxed. Those results are not directly relatable to the 347 CRES diaphragms, but it was believed that the information gained would be useful to this program.

B. SAMPLES

All samples were Type-347 CRES which had been heliarc welded. For all samples, the area analyzed was from the "top" surface immediately adjacent to the weld. The samples analyzed were those described below:

JPL 0981 - welded, no N2H4 (control)

JPL 2005 - welded, in N_2H_4 - 4054 days

JPL 1977 - welded, stressed in N_2H_4 - 4054 days

For samples 2005 and 1977, the areas chosen had been at the liquid/gas interface during the testing. A dark discoloration of interest was particularly obvious in this area.

C. XPS EXAMINATION

All three samples had been rinsed with distilled water upon removal from the hydrazine. Prior to analysis with XPS technique, the samples were further cleaned in an ultrasonic cleaner by serial rinses in trichloroethylene, acetone, and absolute ethanol. Samples were then blown dry with dry nitrogen gas.

The XPS spectrometer (modified HP5950A) averages over a region approximately 1 mm x 5 mm in area and 50-100 Å in depth. Because of the exponential attenuation of the photoelectrons, the immediate atomic surface (approximately 30 Å) was weighted more heavily than the rest.

Except for Na, Zn, and N, al! the observed elements are expected for 347 CRES. (The approximate theoretical composition of 347 CRES is 0.08% C, 2% Mn, 0.05% P, 0.03% S, 1% Si, 18% Cr, 10% Ni, 0.1% Ta, remaining % Fe.) Oxygen is present in the form of various metal oxides and hydroxides as discussed below.

1. Chromium Region

JPL 0981. Cr_2O_3 , CrO_3 , and some reduced Cr^{+3} species at lower binding energy (BE) than CrO_3 , but higher than chromium metal, were observed. chromium hydroxides may also be present.

JPL 2005 and JPL 1977. No differences were observed between these two samples. Spectra were consistent with Cr_2O_3 and chromium hydroxides. The atomic percent of Cr observed increased in the order O_3O_3 C_2O_3 C_2O_3 C_3O_3 #### 2. Fe Region

The atomic percent of iron observed decreased in the order 0981 > 2005 = 1977. For all three samples, effectively Fe₂O₃ and iron hydroxides were observed. There were some differences in the high BE side of the oxide peak. A small amount of FeO was observed on the control (0981) as well as the possibility of low-level FeO or iron sulfides.

3. Mn Region

The atomic percent of manganese observed decreased in the order 0981 $>2005 \approx 1977$. The spectra for all three samples may be assigned to MnO and manganese hydroxides.

Zn Region

The atomic percent of zinc observed decreases in the order 0981 >2005 \approx 1977. Zi.O was present in all three samples.

5. Ni Region

The atomic percent of nickel observed was approximately the same for all three samples, with perhaps slightly more for 2005 as compared to 1977. The Ni was present as ${\rm Ni}_2{\rm O}_3$ and nickel hydroxides. The lower BE on 0981 was probably due to ${\rm Fe}_2{\rm O}_3$.

6. Carbon Region

Approximately the same amount of carbon was observed on all three samples. Primarily aliphatic carbon was present although substantial intensities in the C-0 and C-N regions were observed, showing some differences in detail between the samples.

7. Oxygen Region

Approximately the same amount of oxygen was observed on all samples. The primary peak was due to metal oxides and hydroxides. The lower BE on 0981 was probably due to Fe_2O_3 .

8. Nitrogen Region

The nitrogen intensity increases significantly in the order 0981 <2005

≅ 1977. The primary differences appear on the low binding energy side of 2005 and 1977, where a shoulder characteristic of reduced nitrogen species such as amines and ammonia transition metal complexes was observed. The high binding energy peak was consistent with a variety of species including protonated amines and amide polymers as well as hydrazine salts and nitrites.

D. CHEMICAL ANALYSIS

JPL 1977, stressed

JPL 2003, unstressed

Chemical analysis of propellant and analysis of the decomposition gases indicated no significant differences between stressed and unstressed conditions (Table E-1). Both specimens were exposed to hydrazine for 4054 days.

| | Propel | lant | | |
|----------|------------------------|-------------------|---|--|
| Specimen | Decomposition (wt%/yr) | Dissolved Fe (mg) | N2H2 gas cc x10 ⁻³ day 1 cm ⁻² | |

0.21

0.21

1.48

1.15

0.174

0.154

Table E-1. Summary of Posttest Hydrazine Analysis

E. SEM EXAMINATION OF SPECIMENS

SEM photomicrographs of the surfaces of the specimens indicated that some pitting has occurred at the liquid-vapor interface. As seen in the accompanying photographs, (Figures E-1 to E-4) the distribution of pit sizes varies between the stressed and unstressed specimens. These same samples were sectioned and etched. Photomicrographs of the cross sections revealed no intergranular or intragranular corrosion. The markings seen on the photographs are the result of over-etching.

F. CONCLUSIONS

- The pattern of surface corrosion was similar for each of the specimen examined.
- No intergranular or intragranular corrosion was observed in stressed specimen.

- 3. Fe and Mn dissolved more readily than does Cr, leaving a corroded surface rich in chromium.
- 4. The XPS examination indicated no difference in the chemical nature of stressed and unstressed specimens.
- 5. Only minor differences were observed in metal content or decomposition of propellant between stressed and unstressed configurations.

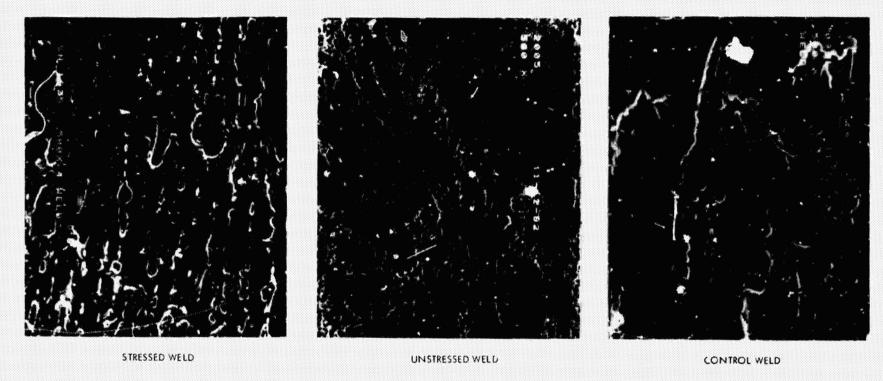
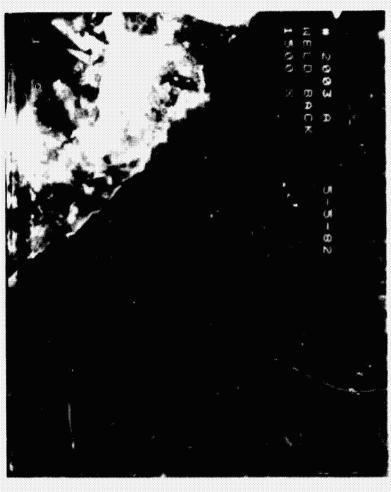


Figure E-1. CRES 347 Weld Specimens, Surface Features at Weld



Figure E-2. CRES 347 Weld Specimens, Surface Features at Heat-Affected Zone

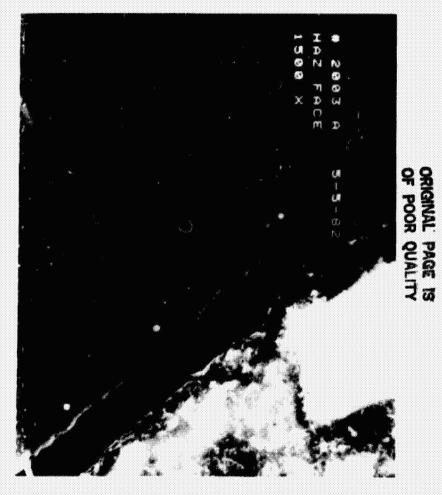




STRESSED WELD-TENSION UNSTRESSED WELD

Figure E-3. CRES 347 Weld Specimens, Cross Sections at Weld





STRESSED HAZ-TENSION

UNSTRESSED HAZ

Figure E-4. CRES 347 Weld Specimens, Cross Sections at Heat-Affected Zone